



TAMPEREEN TEKNILLINEN YLIOPISTO
TAMPERE UNIVERSITY OF TECHNOLOGY

CHAKRA ROKAYA
SOLUTION PROCESSED SCHOTTKY DIODE BASED ON METAL
OXIDES

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ABSTRACT

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Metal oxides are of increasing interest in electronics because of their unique characteristics. They are used as semiconducting interfacial layer between the metal contacts and as active materials in many electrical components like thin film transistor, diodes, and field effect transistors. They have number of advantages like relatively low processing temperature, high carrier mobility, good electrical properties, good transparency, and large-area uniformity. The precursor materials required to make solution processed semiconductor are cheap, easily available in the market, and easy to handle and process.

In this thesis, we attempted to fabricate a Schottky diode based on solution processed metal oxide. The fabricated Schottky diode will be used in a rectifier circuit as low series resistance and high-speed switching device. The input AC signal to rectifier unit will be converted to DC and transfer power to the load circuit. Initially, different metals like aluminum, chromium, copper, gold, silver etc. were studied, analyzed and fabricated on the glass substrate. UV plasma and oxygen plasma surface treatments were explored to enhance film formation properties. Both lateral and vertical structures were studied. Vapor deposition technique was performed for deposition of metal contacts.

The electrical characterization was carried out to find out whether the metal behaves as a Schottky or Ohmic contact to the metal oxide. Lateral and vertical structures were characterized with potentiostat, semiconductor analyzer and temperature stage at different biasing voltages. The measurements shows that all metals behaved more or less as Ohmic contacts. Aluminum and chromium gives Ohmic behavior at all temperatures. Some metals like chromium, copper, silver, gold etc. become injecting when the temperature rises. No true Schottky contact could be identified although this would be predicted according to the energy levels of the semiconductor and the work functions of the metals. Diode were fabricated using a variety of different parameters such as substrate treatment procedures, precursor, sintering method, and multilayer spin coating. The thickness of semiconducting layer and metal deposition thickness also varied during experiment. Unfortunately, no good vertical diodes could be fabricated. Due to the high current, it is believed that the devices are diode short-circuited because of pinholes. This is confirmed by microscopic images showing non-uniform layer, holes, cracks, and other defects.

PREFACE

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CONTENTS

1.	INTRODUCTION	1
2.	BACKGROUND	3
2.1	Solution Processed	3
2.2	Metal-Semiconductor	3
2.2.1	Schottky barrier diode	5
2.2.2	Ohmic Contact	8
2.2.3	Schottky diode I-V Characteristics	10
2.3	Deposition Technique	11
2.4	Application for Schottky Diodes	12
2.4.1	High frequency application	12
2.4.2	Power rectifier:	13
2.4.3	Charge pump	13
2.4.4	Solar application	14
2.4.5	Diode as display driving a circuit	14
3.	EXPERIMENT	16
3.1	Device fabrication steps	16
3.2	Materials	17
3.2.1	Substrate	17
3.2.2	Preparation of precursor	18
3.2.3	Making of thin film on substrate	19
3.3	Electrode materials and Deposition	21
3.4	Device structure	26
3.4.1	Lateral structure	26
3.4.2	Vertical Structure	27
3.4.3	Measurements	28
4.	RESULTS AND DISCUSSION	31
4.1	Semiconductor layer thickness	31
4.2	Metal contacts Current-Voltage analysis for Lateral structure	32
4.2.1	Aluminum	32
4.2.2	Chromium	33
4.2.3	Silver and Gold	35
4.2.4	Copper	36
4.3	Metal contacts Current-Voltage analysis for Vertical Structure	37
4.3.1	Aluminum (Al/IO/Al)	37
4.3.2	Chromium (Al/IO/Cr)	39
4.4	Current-voltage characteristics comparison of indium oxide and indium gallium oxide	40
4.5	Current-voltage characteristics comparison glove box vs room temperature	41

5. CONCLUSIONS	43
REFERENCES.....	44

LIST OF FIGURES

Figure 1.	Schottky diode (a); Metal-Semiconductor junction (b),	5
Figure 2.	Metal and n-type semiconductor, Schottky barrier for $\phi_m > \phi_s$ band diagram before contact (a) ; after contact (b) [18]	6
Figure 3.	Metal and p-type semiconductor, Schottky barrier for $\phi_m < \phi_s$; band diagram before contact (a); after contact b) [18].....	8
Figure 4.	Ohmic metal- semiconductor contact for n- type material $\phi_m < \phi_s$: (a) band diagram before contact ;b) after contact [18].....	9
Figure 5.	Ohmic metal- semiconductor contact for p- type material $\phi_m > \phi_s$: (a) band diagrams before contact ; b) after contact [18]	10
Figure 6.	M-S junction forward bias (a); reverse bias(b) [18].....	10
Figure 7.	E-beam evaporation	12
Figure 8.	Half wave rectifier [19].....	13
Figure 9.	Charge pump circuit [29].....	14
Figure 10.	Steps for Vertical diode fabrication	16
Figure 11.	Steps for lateral structure fabrication	17
Figure 12.	Glass substrate (a) and cleaning agent (b)	18
Figure 13.	Spin coater (a); microscopic view IO layer, silicon(b); glass(c)	21 22
Figure 14.	Compilation of valance and conduction bands for transition metal oxides(a);Organic semiconductor on an absolute scale (b) [37]	22
Figure 15.	Vapor deposition chamber	23
Figure 16.	Vapor deposition; 0.4 M IO (a), 0.2M IO lateral (b), 0.2 M IO vertical (c)	24
Figure 17.	Lateral structure (a) ;Microscopic images of lateral contact(b)	26
Figure 18.	Vertical structure(a), microscopic view of diode(b), fabricated diode(c).....	27
Figure 19.	Measuring unit; Keysight(a,b) and Zahner(c,d).....	30
Figure 20.	Thickness vs RPM.....	32
Figure 21.	I-V Characteristics of Aluminum(a, b)	33
Figure 22.	Temperature dependence and I-V characteristics of Chromium (a, b).....	34
Figure 23.	Temperature dependence and I-V characteristics : Silver(a), Gold(b)	36
Figure 24.	Temperature dependence and I-V characterstics of copper.....	37
Figure 25.	Vertical structure and I-V characteristics of Al/IO/Al	38
Figure 26.	Vertical structure (a), I-V characteristics (b).....	39
Figure 27.	I-V characteristics IO and IGO.....	40
Figure 28.	I-V characteristics inside glovebox and outside area	41

LIST OF SYMBOLS AND ABBREVIATIONS

A	Diode Area
AC	Alternating current
Å	Angstrom
C	Capacitance
d	Thickness of semiconductor
DC	Direct Current
E_c	Conduction band energy
E_f	Fermi level energy
E_{vac}	Vacuum level energy
E_v	Valance band energy
I	Diode current
I_o	Reverse saturation current
IO	Indium Oxide
IGO	Indium gallium Oxide
IZO	Indium Zinc Oxide
J	Current density
k	Boltzman's constant
K	Kelvin
η	Ideality factor
MS	Metal - Semiconductor
V	Voltage
V_{IN}	Input Voltage
V_{DC}	Output Voltage
V_f	Forward voltage
V_r	Reverse voltage
V_o	Potential difference
W	depletion width
q	Elementary charge
R	Resistance
ϕ_b	Schottky barrier energy
ϕ_m	Work function of metal
T	Temperature
t_T	Transient time
R_L	Resistor
TFT	Thin film transistor
W	Depletion width
χ	Electron affinity
ϵ_0	Vacuum permittivity
ϵ_r	Relative permittivity

1. INTRODUCTION

Metal oxide semiconductors such as indium oxide (IO), indium gallium zinc oxide (IGZO) are playing important role in day-to-day electronics. They have a number of advantages like superior electrical properties such as higher carrier mobility, optical transparency, large- area uniformity, low processing temperature etc. [1] [2] There are many ways for the fabrication of the diode for example vacuum sputtering, vapor deposition, photolithography etc. Different printing techniques might use in case of organic metal oxide like screen or flexography that optimize the cost and print over large area. As the metal oxide are lower temperature cured, this helps to make diode in plastic or paper substrate more flexible. [2] Surface properties has major role for performance of the device. The high quality of achievement of the semiconductor layer was obtained by adjusting layer in a controlled manner. This helps to remove impure defects balancing the fermi level energy. [3]

Indium oxide thin films have large value of single crystal mobility up to $230\text{cm}^2/\text{Vs}$ and controllable conductive ranging from semi-insulating to highly conductive have favorable bulk transport properties.[2] However, solution processed indium oxide thin films provide mobility's around $10\text{cm}^2/\text{Vs}$. It was observed that fabrication method using roll-to-roll print for TFT using solution processed indium oxide has provide mobility up to $8\text{cm}^2/\text{Vs}$. [4] Indium oxide has band gap of 3.6 eV - 3.75 eV and indium gallium oxide with band gap of 4.9eV . [5] Band gap of indium gallium oxide can varied and depends on doping concentration or chemical composition. [Yang, C. 2017] In TFT fabrication, for variation of indium gallium contents in the four different samples gives band gap of 4.5 , 4.2 , 4.0 , and 3.5 eV respectively [6]. Therefore, band gap of sample may varies. Valance band is lowest unoccupied orbital of semiconductor and conduction band is highest occupied orbital. Metal oxide layer is used as an active conductive layer for the manufacturing the thin film transistor and diodes. [7]

Like the transistor, a diode is also a basic building block in many circuits. It is used in many thin film or transparent electronic applications, e.g. radio frequency tags (RFID), solar cells, amplifiers, photo detectors, transparent display, sensors, amplifier, logic gates and high quality Schottky diode. [1] [2] There has been lots of experiment and results done for fabrication of TFT, modeling, understanding charge trapping, but only few on Schottky diodes based on metal oxide. Therefore, there has been the interest for fabricating Schottky diode and analyzing its electrical properties like rectifications ratio, current density, barrier height, high reverse break down voltage. At metal-semiconductor interfaces, Schottky barrier are formed which is important region of Schottky diode. The factor that affect and influence the efficiency of Schottky device is called ideality factor. Ideality factor denoted as η . Current-voltage analysis and capacitance-voltage analysis used for measuring barrier height [8].

In this thesis, Schottky diode fabrication some parts was done at room temperature like thin film deposition using spin coating and other hand, deposited layer were around temperature of 300°C. Film deposited on glass substrate and in future deposition is possible flexible PET or plastic. Other things included like making the precursor of indium nitrate hydrate, spin coating for film, main purpose of diode fabrication is to use in low series resistance and high frequency application. E-beam vapor deposition (Ohmic and Schottky contact) in high vacuum with pressure less than 2×10^{-6} bar at deposition rate 3Å/s. Ohmic contact gives linear relationship between current and voltages where as Schottky has non-linear, asymmetrical due to a barrier between metal semiconductor junction. [9] Based on the experimental work, study different metal whether Ohmic or Schottky and current voltage characteristics were studied. Temperature dependence measurements performed and compare for the lateral structures. In order to get the good results, lots of experiments were performed that includes changing the parameter like semiconductor layer thickness for different structure, spin coating speed, sintering temperature, UV/oxygen plasma treatment, fabrication of multi layers to avoid pinholes, change in molar concentration, various treatment to the substrate, vapor deposition parameter, rate etc.

In Chapter 2, it describe the metal-semiconductor junctions. It explains the Schottky diode, and formation of barrier between metal semiconductor junctions. It also describe the properties and its applications. This chapter also included the voltage-current characteristics of the Schottky diode and deposition technique of the metal electrode for conduction.

In chapter 3, it describe the different diode fabrication steps. Finally, chapter 4 gives the conclusions and gives recommendation for future research.

2. BACKGROUND

2.1 Solution Processed

Metal oxides are dissolve in solution used in wide range of applications for manufacturing the high performance diodes as well as thin film transistors. Metals such as Indium, Gallium, and Zinc have recently gained momentum because of their several advantages in electronic components design and high carrier mobility, stability, good optical transparency and low processing temperature. [1]

There is a cheap, simple method for making sol-gel. This method includes gallium nitrate hydrate, indium nitrate hydrate, zinc nitrate hydrate dissolved in two-meth-oxy ethanol, stirred more than 7 hours at temperature of 70°C.

2.2 Metal-Semiconductor

A device with two terminal is diode. The resistance of diode is high in reverse direction. On the other hand, it has low resistance in forward direction. A normal PN diode has junction formed between p- type and n-type semiconductor. Holes are a majority carrier in p-type and electrons are majority carrier in n-type. Metal-semiconductor diode is different from p-n diode. M-S diode has junction between metal contact and either n or p-type semiconductor.

The junction formed between metal and semiconductor having unequal work function called Metal-Semiconductor junction (M-S). The metal-semiconductor junction may two types non-rectifying or rectifying. Non-rectifying metal-semiconductor junction is Ohmic contact. Similarly, Schottky is a rectifying metal-semiconductor junction. [10]

Majority carrier is responsible for the current flowing through the junction. Mechanism for the flow of current are three types. Those are diffusion, tunneling and thermionic emission. Described below is different mechanism for current across M-S junction.[11]

1. $I_{\text{diffusion}}$ is the diffusion current due to concentration gradient and diffusion current density. [12]Diffusion results in formation of depletion layer which is large enough. [11]The resulting current density equals :

$$J_n = \frac{q^2 D_n N_c}{V_t} \sqrt{\frac{2q(\phi - V_a) N_d}{\epsilon_s}} \exp\left(-\frac{\phi_b}{V_t}\right) \left[\exp\left(\frac{V_a}{V_t}\right) - 1\right] \text{----- (1)}$$

When the voltage is applied to the M-S junction, there is an exponential rise of Current. The current depends on applied voltage V_a and barrier height. Further, Formula for J_n was given below.

$$J_n = q\mu_n\varepsilon_{max}N_c \exp\left(-\frac{\phi_b}{V_t}\right) \left[\exp\left(\frac{V_a}{V_t}\right) - 1\right] \text{-----} (2)$$

$$\text{where, } \varepsilon_{max} = \sqrt{\frac{2q(\phi - V_a)N_d}{\varepsilon_s}}$$

2. $I_{\text{tunneling}}$: The electric current caused by tunneling through the junction barrier. [12]. The multiplication of velocity, density and charge carrier are responsible for tunneling current. The velocity of average number of carriers close to the barrier is similar to the Richardson expression for velocity. The product of probability of carriers to tunnel and number of available electrons should matches to carrier density. The expression for the current density is given below: Θ , yielding:

$$J_n = qv_R n \Theta \text{-----} (3)$$

where q is the charge, Θ is tunneling probability and v_R is Richardson velocity, where the tunneling probability is obtained from:

$$\Theta = \exp\left(-\frac{4}{3} \frac{\sqrt{2qm^*}}{h} \frac{\phi_B^{\frac{3}{2}}}{\varepsilon}\right)$$

In addition, the electric field equals $\mathcal{E} = \phi_B/L$, where m^* is electron mass, ϕ_b is barrier height, h is planks constant. From the equation above, it can be seen that the tunneling current is related to the M-S junction barrier height and rise exponentially.

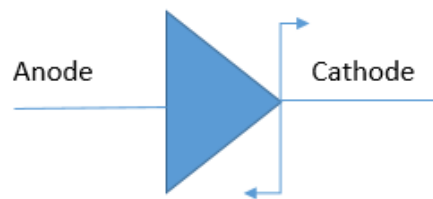
3. $I_{\text{Thermionic emission}}$ is current due to thermionic emission. Thermal energy or temperature is responsible for the injection of electron. If the thermal energy is high enough then the electrons can cross the M-S barrier resulting current in the junction. [11] The equational expression for the current is given by equation below:

$$J_{MS} = A^* T^2 e^{-\phi_b/V_t} \left(e^{\frac{V_a}{V_t}} - 1\right) \dots \dots \dots (4)$$

Where $A^* = \frac{4\pi q m^* k^2}{h^3}$, A^* is Richardson constant, T is absolute temperature.

2.2.1 Schottky barrier diode

Schottky diode is named after Walter H. Schottky. It is an unilateral device. The Schottky diode is known as surface barrier diode, majority carrier device, hot electron diode. The forward voltage drop is very low. [10] [12] It has also high-speed action. Working in range of megahertz to gigahertz because of fast response time. In addition, at high frequency about 50 Mhz rectification had been achieved for printed diodes. [13] MIM diode is superior to Schottky diode; junction area is in Nano scale range, operates in THz frequency. In addition, in MIM diode had thin insulating layer sandwiched between two metal. [14] A metal-semiconductor junction, creating a barrier and Schottky contact at metal-semiconductor interfaces as shown in figure 1. The barrier is responsible for potential energy barrier for electrons at junction. The most common metal used for barriers are silver, gold, platinum, chromium, tungsten, molybdenum and N-type silicon acts as the semiconductor to be used. [12] Generally, metal is a positive electrode and N- silicon is negative electrode of the diode.



(a)



(b)

Figure 1. Schottky diode (a); Metal-Semiconductor junction (b),

The minimum amount of energy required to eliminate the electron bounded to the solid to vacuum level is define as work function. Fermi level and vacuum level have their own energy. The difference in energy between them gives work function. To metal, it is indicated as $q\phi_m$ and furthermore to semiconductor it indicated as $q\phi_s$. If the energy of $q\phi_m$ is

applied then the electron released freely from the surface of the metal. Vacuum level is defined as an energy range where an electron is free which is no longer bound to an atomic nucleus and has zero kinetic energy. [15] Fermi level describes the electron potential or electrostatic energy at absolute zero temperature. "Fermi Sea" of electron observed at absolute zero where electron pack into the lowest available energy states. [15] [16]

The positive charges generated on it, when the negative charges brought near to the metal surface. [17] An applied electric field and image force combined that reduce the effective work function. This result in decreasing the barrier height, called Schottky effect. The metal semiconductor rectifying contacts are achieved called Schottky diode. [18]

As shown in figure 2, the metal have work function $q\phi_m$ and the semiconductor have work function $q\phi_s$. When metal and semiconductor are brought in contact together, there will be transfer of charge between them unless fermi level will come at same level. This means fermi level of metal and semiconductor are equal. In the case, ϕ_m is greater than ϕ_s , fermi level of metal is lower than that of semiconductor before they are made in touch with each other. The rise in electrostatic potential of semiconductor is required to align the metal and semiconductor fermi level. The metal electron energy must be increase than semiconductor. The charge flow causes the semiconductor bands to bend upwards. W is the region of depletion, formed at the junction. The negative energy that created on the metal energy matches the positive charge inside the depletion width [15] [19]

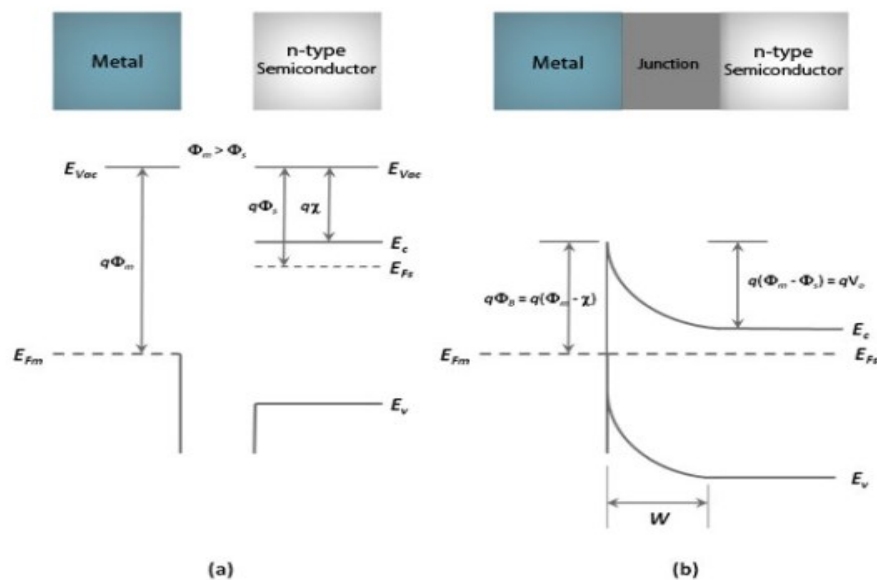


Figure 2. Metal and n-type semiconductor, Schottky barrier for $\phi_m > \phi_s$ band diagram before contact (a) ; after contact (b) [18]

$$\text{Depletion width, } W = \sqrt{\frac{2\epsilon_0\epsilon_r(V_0-V)}{qN_A}} \dots\dots\dots(5)$$

Where, ϵ_r is semiconductor permittivity, ϵ_0 is the vacuum permittivity, V_0 is a developed potential, V is the external voltage, N_A is the dopants concentration, q is the charge. [12]

Equilibrium contact potential V_0 is difference in energy level of work function related to the metal and semiconductor. This potential stop more diffusion of electron between the metal and semiconductor's conduction band. Expression for the equilibrium contact potential is given below.

$$V_0 = \phi_m - \phi_s \dots\dots\dots (6)$$

The Schottky barrier height $q\phi_b$ is defined as the difference between the metal work function and edge of the semiconductor conduction band [19] [20] and is expressed as

$$q\phi_b = q(\phi_m - \chi) \dots\dots\dots (7)$$

Where $q\chi$ is defined as electron affinity, the difference between energy level of vacuum to conduction band edge of semiconductor. When the forward and reverse voltage applied to the junction, contact potential value either raised up or lowered down in the junction.

As shown in figure 3, the metal and p- type semiconductor with $\phi_m < \phi_s$, in order to achieve the equilibrium state of fermi level, metal side should be positively charged semiconductor should negatively charge. The charge flow causes the semiconductor band to bend downwards and a thin depletion region W . [19] [21] The negative charges due to ionized acceptors within matches the positive charge on metal.

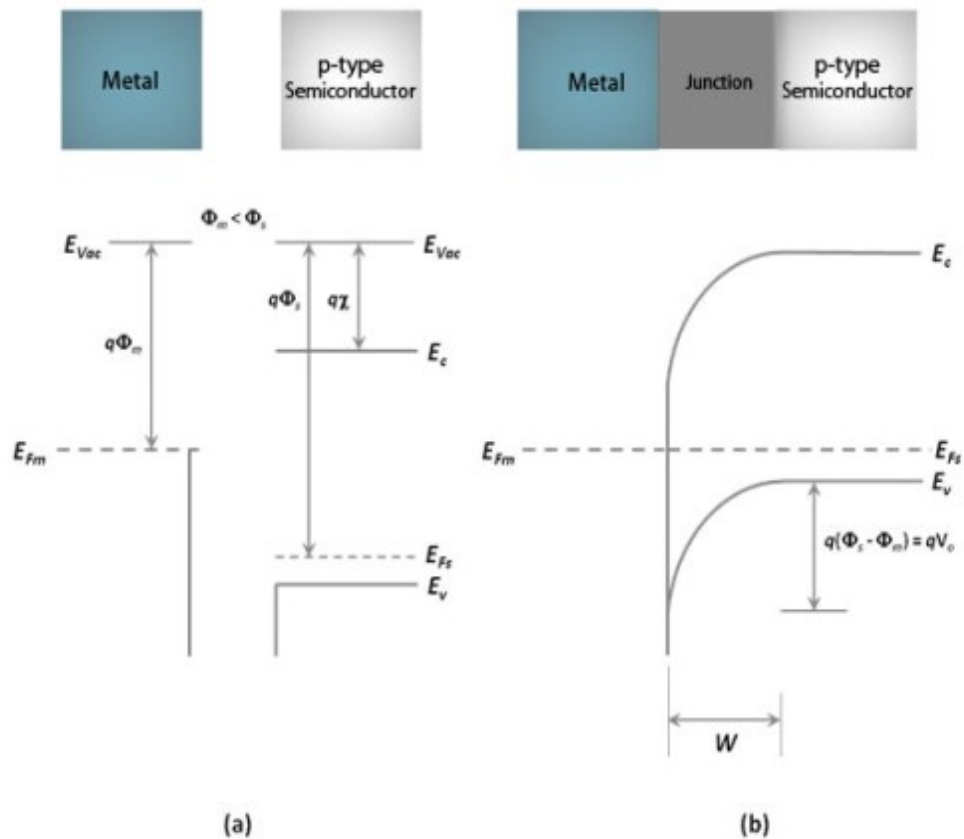


Figure 3. Metal and p-type semiconductor, Schottky barrier for $\phi_m < \phi_s$; band diagram before contact (a); after contact (b) [18]

2.2.2 Ohmic Contact

Ohmic contact shows linear I-V characteristics either forward bias or reverse bias. In this contact, metal and semiconductors brought together. During biasing current flow in and out of junction and there is no any loss and depletion region between metal and semiconductor. [15]

For the ideal metal-semiconductor, the fermi level is in equilibrium and aligned by majority carriers when the charge induced to the contact. For figure 4: for $\phi_m < \phi_s$, n-type case, there will be the migration of electrons from one junction metal to another junction semiconductor until the fermi level and thermal equilibrium is aligned between them. This causes the semiconductor electron energy increased comparing the electron energy of metal. There is a small barrier between metal and semiconductor junction. So, by applying a small voltage to the M-S junction, electrons can easily cross the junction region causing flow of current through the junction. [18]

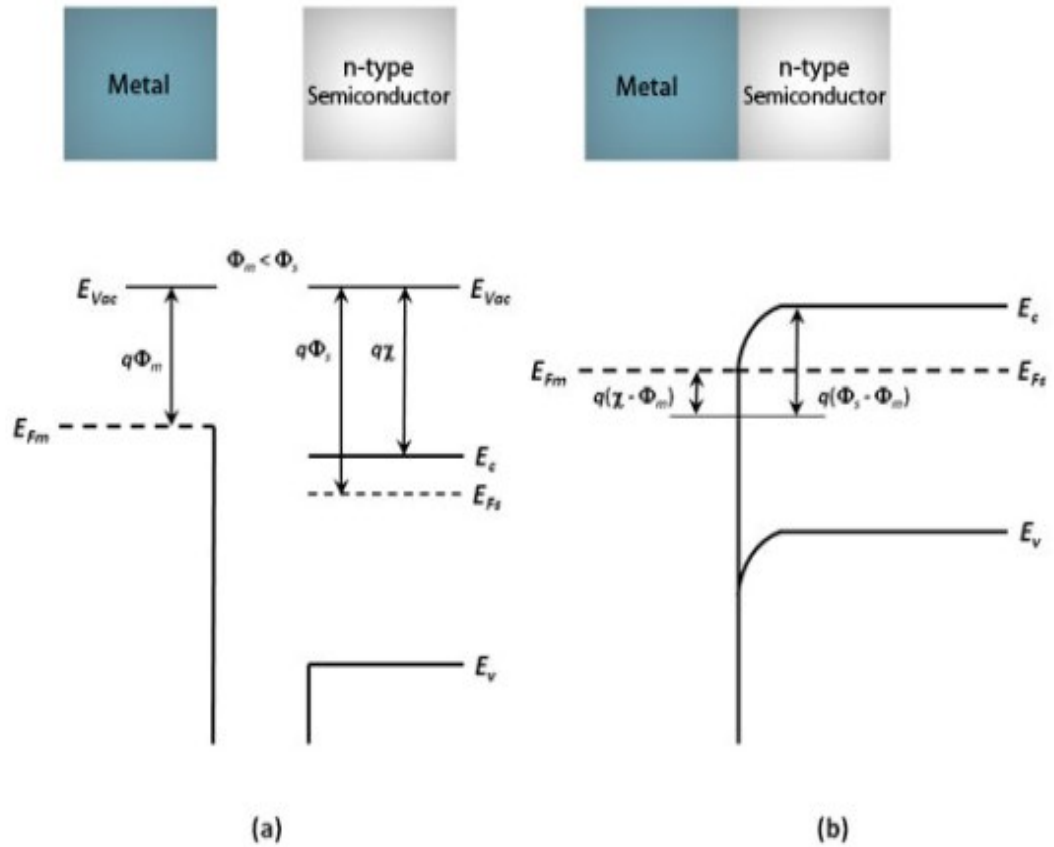


Figure 4. Ohmic metal- semiconductor contact for n- type material $\phi_m < \phi_s$:
(a) band diagram before contact ;b) after contact [18]

Similarly, for p-type semiconductor $\phi_m > \phi_s$, the hole can easily flow across the junction until the fermi level are aligned. No depletion region occurs in the semiconductor like that of Schottky contact. As shown in figure 5: the band diagram related to energy of the p-type Ohmic contact.

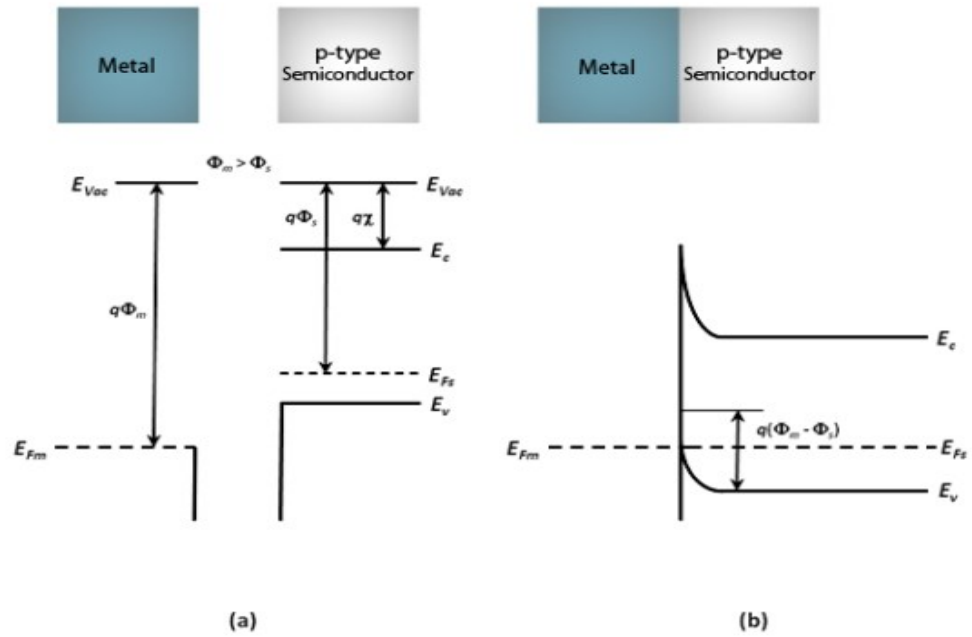


Figure 5. Ohmic metal- semiconductor contact for p- type material $\phi_m > \phi_s$,: (a) band diagrams before contact ; b) after contact [18]

2.2.3 Schottky diode I-V Characteristics

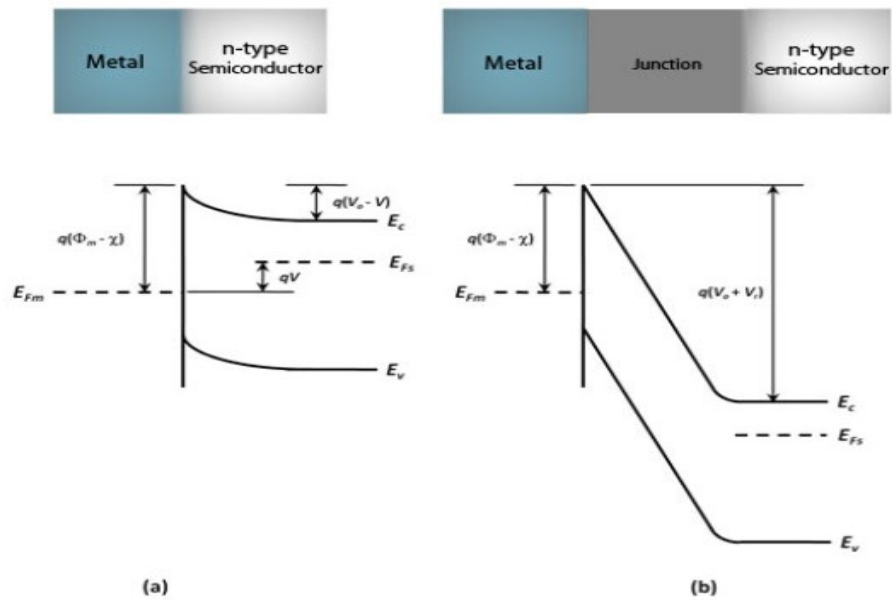


Figure 6. M-S junction forward bias (a); reverse bias(b) [18]

When the biasing voltage applied across it, the current starts to flow in the diode. The diode can be either forward bias or reverse bias. During forward bias of Schottky diode in figure 6 (a), an initial contact potential was V_o , which after biasing decreases to $V_o - V$. Hence, the diffusion of an electron in a semiconductor band occurs through the depletion region near the metal. This causes the electrons in a semiconductor band to diffuse across the depletion region to the metal. From metal to semiconductor junction, there will be the flow of carrier that results in forward current. [18] [21]

Similarly, during negative biasing of the Schottky diode, barrier height becomes the sum of reverse voltage and contact potential. This means barrier height becomes large. Hence, there will be no reverse current flow. From the figure 6, whether the diode is forward bias or reverse bias, the barrier slows the electron that are passing through the metal-semiconductor junction. The expression for Schottky diode equation matches to a p-n diode. [18]

$$I = (I_0 e^{\frac{qV}{kT}} - 1) \dots \dots \dots (8)$$

Where, $I_0 = ABT^2 e^{-q\phi_b/kT}$

In above equations, I_0 current in reverse, V is the external input voltage, K is the Boltzmann's constant, T denotes temperature and q denotes electron charge, A is the area of Schottky contact, B is a constant parameters, ϕ_b is the Schottky barrier height. In this case, when the majority carriers are injected from a semiconductor junction to the metal junction then it results in forward current and there is no reverse current. Minority carrier injection is not present and delay time is negligible in Schottky diode. [1]

2.3 Deposition Technique

Thermal evaporation is one of the most common physical vapor deposition technique for the metal or thin film on the substrates. First, step is selection of the desired mask or pattern. Then, attachment of sample with mask. Samples are place invert position in sample holder. The chamber consists of different metals like gold, silver, chromium, aluminum etc. the metals placed at lower part of chamber. Generally, vacuum inside the chamber is very high. Further, selection of desired metal. Followed by heating with the electron beam gun, until it gets melting and vaporized. The metal vapor rises up and resulting deposition metal sample attached to the sample holder. [22]

Physical Vapor deposition methods include electron beam, sputtering, thermal and plasma arc. The one of the most common source the heat is Electron beam or E-beam evaporation. Very high voltage electricity used to heat the material heated by high electric voltage usually 10KV at high pressure. Electron beam is main source of high-speed electron, which strikes the metal. It is small size. It has a filament, which is very hot. The beam strikes on the crucibles, which consists of material to be melt and deposit. [23] The material was heated with high power and is evaporated and deposited on the substrate. [24]

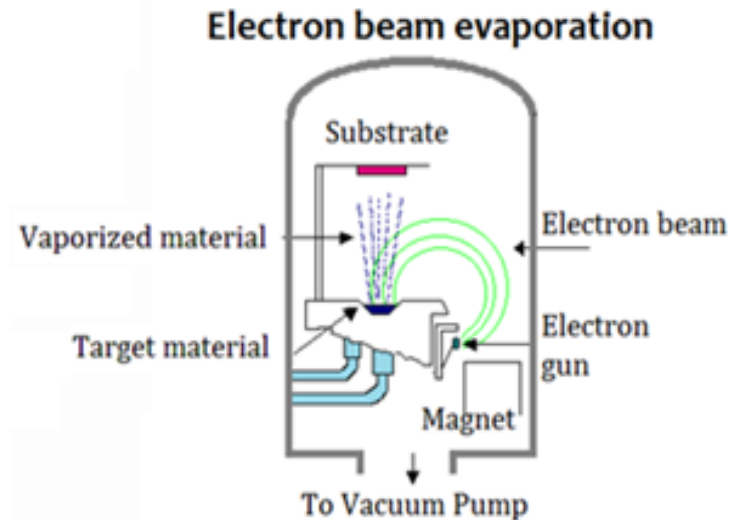


Figure 7. E-beam evaporation

2.4 Application for Schottky Diodes

There are many areas where Schottky barrier diodes are widely used. They have unique properties where a normal diode is replaced by Schottky diode and give good range of performance. In high-speed application, mixers, detectors, monitoring short pulses etc. Schottky diodes are used. The following sections describe its applications. [25]

2.4.1 High frequency application

Schottky diode is widely used in radio frequency application because they have fast on/off speed. Diode ring mixer use Schottky diode in high performance work. Schottky diode forward voltage drop is very low around 0.1 to 0.45V and low capacitance. These features helps to act as RF detectors. [11]

Especially in RFID, tag Schottky diode role for large frequency rectification. The diode maximum frequency is dependent on the transit time (t_T). It is the time for the charge to move from one electrode top another. The expression for maximum frequency equation is: [13]

$$f_{max} = \frac{1}{t_T} = \frac{\mu(V_{IN} - V_{DC})}{d^2} \dots \dots \dots (9)$$

Where, the V_{IN} is the voltage applied as input, V_{DC} is the voltage as output and d is the semiconductor thickness.

For RF applications capacitance has its own great importance, thinking Schottky diode as parallel plate capacitor. Doping profile and physical dimension determines capacitance of the parallel plate. Depending in applied voltages, in forward the thickness of junction will decrease and in reverse, it will increase. Therefore, junction capacitance limits the switching speed. [10] The capacitance equation is given below.

$$C = \frac{\epsilon_r \epsilon_0 A}{d} \quad \text{----- (10) Where A is Area}$$

of diode, d is semiconductor thickness, ϵ_0 is vacuum permittivity and ϵ_r is relative permittivity.

2.4.2 Power rectifier:

Schottky barrier diodes are used to convert AC to DC in large electrical-power areas, as a half wave, full wave or bridge rectifier. As Schottky, diode have very less drop voltage around diode and density of current is high. As a result, low power is waste than that of PN diode. Figure 8 shows a simple half wave rectifier circuit. It consists of a diode in series with capacitor, which is in parallel with load resistor R_L . V_{in} , the input voltage and V_{out} is the output DC voltage after rectification. [19] [21]

During a positive half cycle, the diode is on and conducting that results charging of capacitor and power delivered to load. On the other hand, during a negative cycle diode is not conductive. For examples: RFID high frequency application uses diode with high DC output based on input AC voltage. [19]

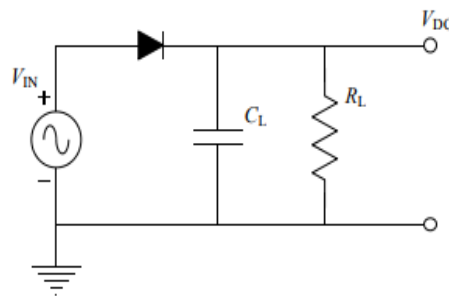


Figure 8. Half wave rectifier [19]

2.4.3 Charge pump

A charge pump circuit converts AC signal into DC. For the design of the circuits, especially Schottky diodes and capacitors are used. They are arranged in many stages either 3-stages or 4-stages. [26] This circuit have variable input, high gain, efficiency and small

cost for design. [27] This circuit helps in energy harvesting generally for RF signals especially in RFID. The charge pump circuits had advantages than the half wave rectifier. The output DC voltage obtain from charge pump circuit is higher than the rectifier. [28]

Design of the circuit can be varied and flexible depending on the capacity of diode and value of the capacitor. This full fill the requirements of the charge pump output voltage. For examples, the value of diode and capacitor should be high, if the charge pump needs to operate at low frequencies. [28]

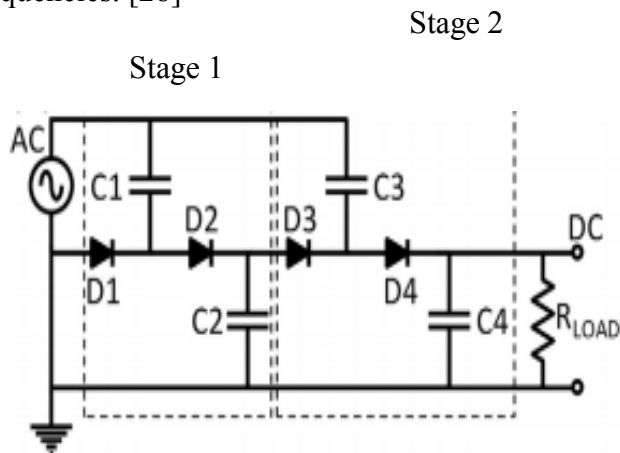


Figure 9. Charge pump circuit [29]

The expression for the output voltage of charge pump circuit is:

$$V_{out} = [NV_{IN} - V_F * 2(N - 1) - (a)] \dots \dots \dots (10)$$

Where N is the number of stages, V_{IN} is the input Voltage and V_F is the turn on voltage of each diode, denotes other power losses. [30] [26]

2.4.4 Solar application

Sun is the power source of the energy. Solar panel generate photovoltaic energy and use to charge the batteries. Schottky diode connection with solar cell circuitry for the protection from the negative biasing that leads to damage of the circuit. Furthermore, the efficiency of the solar cell device will reduce due to voltage drop. The Schottky diode with low voltage drop are useful to maintain voltage in this application.

2.4.5 Diode as display driving a circuit

The electronic paper display are being popular in many electronics applications LCD, OLED because of optical properties, less consumption of power, more flexible, robustness etc. [31] Fabrication of non-linear device such as Schottky diode or p-n diode on transparent substrate like glass using sputtering, vapor deposition technique. Printing

techniques (screen or gravure) applied for the electrode deposition of elements required for the display system. [32]

Display driving circuit handles the control and operation of switching action on display. Each display segments have their own address. A display division into rows and columns because it helps to address each pixel directly and make more practical. There are two types of matrix used passive matrix and active matrix. Active matrix provides benefits over passive matrix. Passive matrix have slow response time, less efficient, drive current is high for OLEDs. [31]

3. EXPERIMENT

This chapter describes the experimental work like substrate used, making of sol gel precursor as semiconducting layer, study of metal rectifying contact or Ohmic contact, fabrication process and diode structure etc.

3.1 Device fabrication steps

The first steps involves cleaning of the glass substrate. The cleaning agents used are acetone, isopropanol and deionized water. After that deposition of the metal on the glass as base electrode. Vapor deposition technique facilitates the deposition of metal. The section 3.2 describes more in details .Then, take out the sample from the chamber and spin coat the metal oxide layer on it around 2000 rpm. Then, sintering at 300 °C for 30 minutes. Finally, then the layer is setup then again deposit of top electrode metal by vapor deposition technique [33].

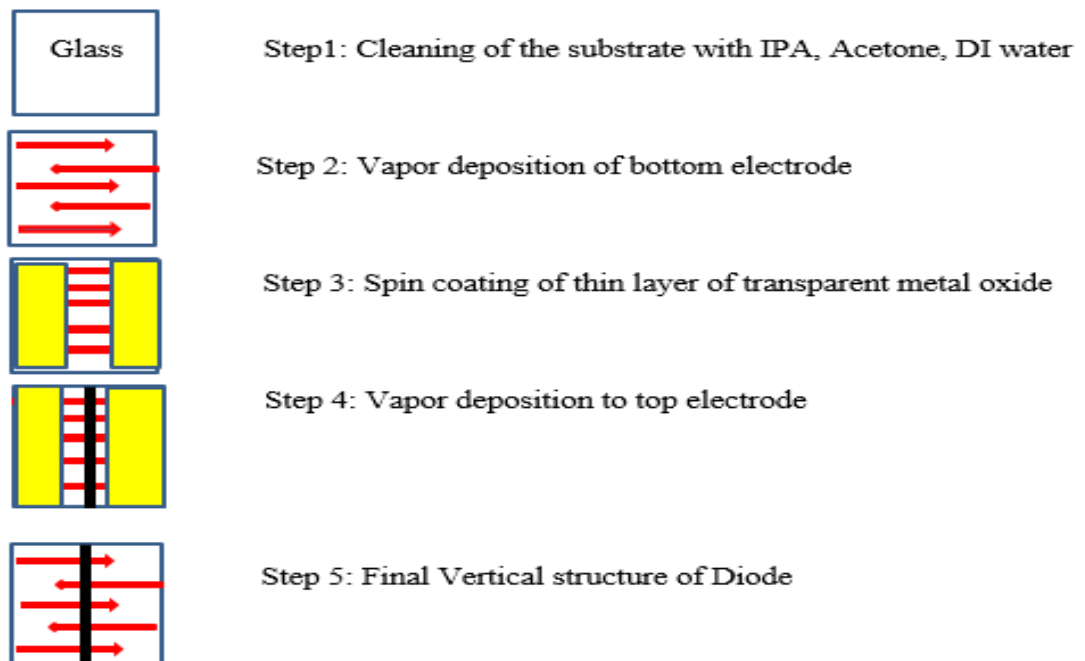


Figure 10. Steps for Vertical diode fabrication

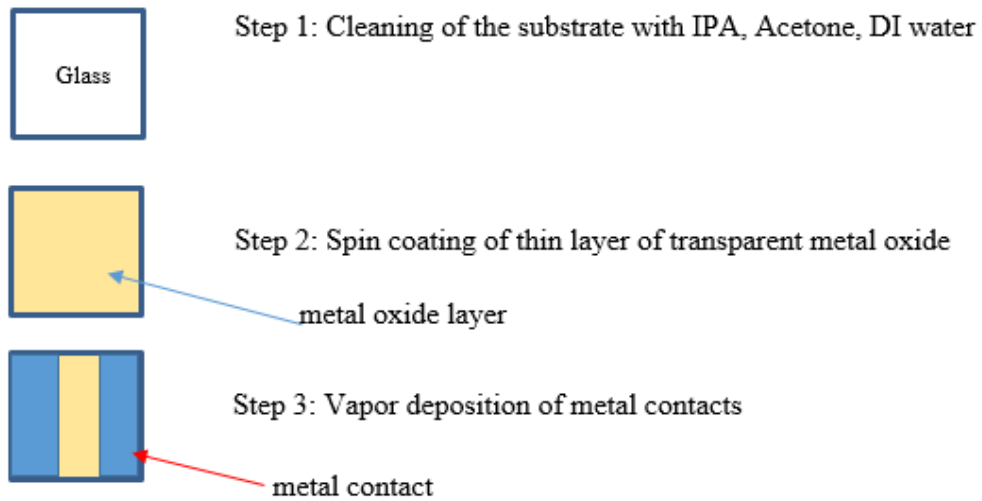


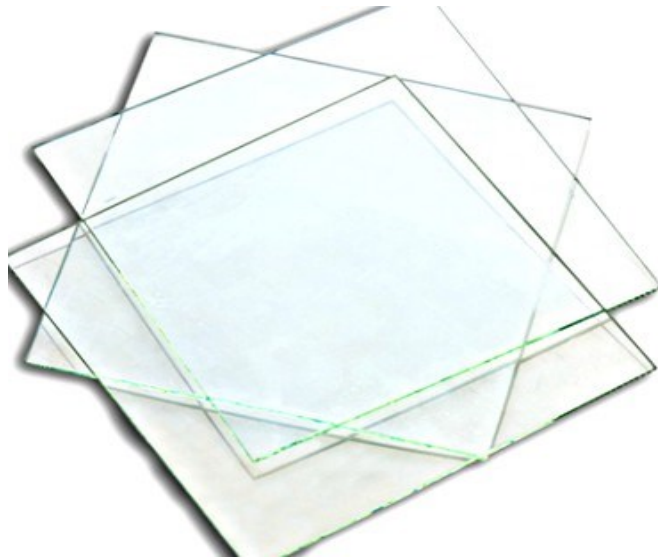
Figure 11. Steps for lateral structure fabrication

3.2 Materials

3.2.1 Substrate

Substrate is a surface material for deposition of semiconducting layer or the metal. As a substrate, silicon wafer initially used for few experiments. Later, glass replaced silicon and used substrate.

There are many advantages of glass that was used a substrate. It has high transparency, surface smoothness and uniform area, and heat stability, thermal and other mechanical properties. In our experiment the temperature, goes up to 300°C, and the substrate can sustain higher than this temperature. Before the deposition of the semiconducting layer, the substrate should undergo substrate treatment with isopropanol, acetone, ionized water, UV or oxygen plasma treatment. Those treatments remove dirt particles and make surface smooth. Flexible substrate like PET may act as substrate in future. [28]



(a)



(b)

Figure 12. Glass substrate (a) and cleaning agent (b)

3.2.2 Preparation of precursor

Indium (III) Nitrate hydrate ($\text{In}(\text{NO}_3)_3 \cdot x\text{H}_2\text{O}$) and Gallium Nitrate Hydrate $\text{Ga}(\text{NO}_3)_3 \cdot x\text{H}_2\text{O}$ from the manufacturer Sigma-Aldrich (99.9%) were used for making semiconducting layer. $\text{In}(\text{NO}_3)_3 \cdot x\text{H}_2\text{O}$ and 5% of Gallium Nitrate Hydrate $\text{Ga}(\text{NO}_3)_3 \cdot x\text{H}_2\text{O}$ dissolved with 2-methoxy ethanol. This results in 0.2 Molar solution. Then, a solution stirred for 12 hours at the temperature of 70°C . The 0.2 Molar precursor is transparent.

Sol-gel precursors were made, indium nitrate hydrate and indium gallium nitrate hydrate. Sol-gel gives a semiconducting layer of indium oxide and indium gallium oxide. Five percent gallium was used as a dopant, increasing impurities, extrinsic results in higher mobility.

The 0.2 M and 0.4M sol gel were prepared for the making semiconducting layer.

3.2.2.1 Methods of preparation

1 Molar solution is 1 mole of substance dissolved in 1 liter. [34]

The molecular weight of Indium nitrate hydrate, $(\text{In}(\text{NO}_3)_3 \cdot x\text{H}_2\text{O})$ is 300.83 gm.

1 M of $[\text{In}(\text{NO}_3)_3 \cdot x\text{H}_2\text{O}] = 300.83 \text{ gm/liter}$

$$\begin{aligned} 0.2 \text{ M of } [\text{In}(\text{NO}_3)_3 \cdot x\text{H}_2\text{O}] &= (300.83 * 0.2) \frac{\text{gm}}{\text{liter}} \\ &= 60.166 \frac{\text{gm}}{\text{liter}} \end{aligned}$$

It is not more practical to make large volume of solution i.e. in 1 liter, so just making 0.2 M around 10 milliliter (ml).

$$0.2\text{M in } 10 \text{ ml} = (60.166 * \frac{10}{1000}) = 0.601 \frac{\text{gm}}{10 \text{ mlliter}}$$

Hence, 0.601 indium nitrate hydrate dissolved in 10 ml solution.

Similarly, in case of indium gallium oxide, 5% of it was added to the previous solution and similar calculation was repeated. [35]

3.2.3 Making of thin film on substrate

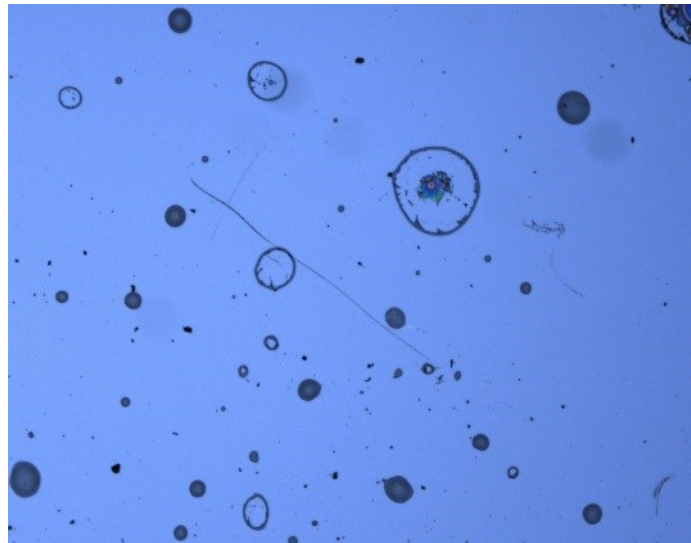
Spin coating is the technique for the deposition of the thin film. The different thickness of semiconducting layer is obtained depending on the speed of the spin coater. The RPM varies from 1000 to 6000. Increasing in speed results in lower thin film. The minimum thickness was 20.82nm at 6000 rpm and the maximum thickness was 45.466 nm at 1000 rpm. Deposition of IO and IGO on the silicon and glass substrate. Profilometer measured the thickness of the film. The spin coating was not uniform and some island and hole structures observed from the microscope.

Once the thin film is spin coated on the glass. The next two steps are heat treatment. The first is 90°C dry heating of the samples for 15 minutes. The second step is 300°C sintering of the sample for 30 minutes. Sintering is an annealing as well as heating process of the sample to meet its specifications. Sintering temperature is below the melting point of the

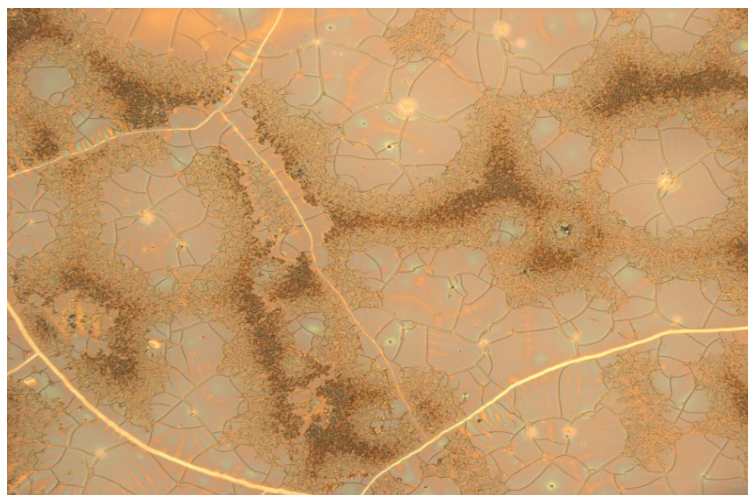
required phase. Sintering process removes unnecessary defects, improves the quality of sample, improves ductility etc.



(a)



(b)



(c)

Figure 13. Spin coater (a); microscopic view IO layer, silicon(b); glass(c)

3.3 Electrode materials and Deposition

E-beam was a vapor deposition technique for the deposition of the metal contact. Depending on the metal and their work function, desired metals chosen for the fabrication process. Metal oxides with wide range of work function make it possible to involve in large area application. Energy band is the difference between energy level between valance band and conduction band in semiconductor. This level vary depending on zero bias, forward bias and reverse bias of metal-semiconductor junction. The valance band edges is around 1 to 3eV in most of metal oxides which is below H₂O oxidation potential. Similarly, energies for conduction band edges are near to H₂O reduction potential. In aqueous solution, oxide minerals indium nitrate hydrate are strong photo-oxidation catalyst. [36] Indium oxide is wide band n-type semiconductor, valance band off set in the range 2.6 to 2.9 eV [37][Chang T. 2015] Co-sputtering was done for deposition of a-IGO, the band gap between valance band and conduction band is around ~3.6eV when photon energy $\geq 2.5\text{eV}$. [7] The energy level diagram metal oxide and organic interface shown in figure 14. Metal oxide may be electron injecting (TiO₂, ZrO₂) or holes injecting (CUO, NIO, IO). Energy level alignment is necessary to use this material at highest potential. As, the different metals have different work function. The work function determines the positions of oxide conduction and balance band comparing to vacuum level. Charge injection barriers determined from the difference in metal work function and HOMO and LUMO energy levels. [37] Metal oxide have wide range of work function from ~2 eV to ~7 eV. Figure 15 shows oxide energy level diagram. UPS spectra helps to determine the band edges and band gaps studied from literature values [36].

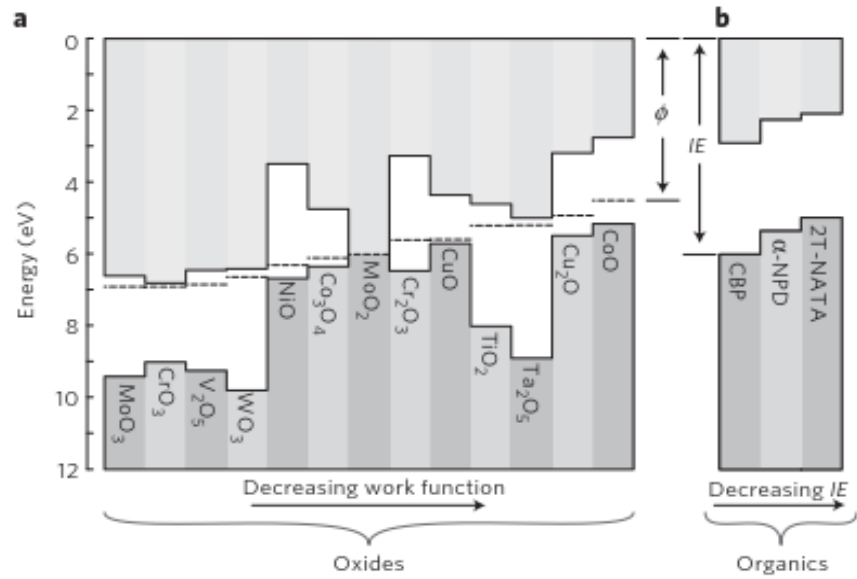


Figure 14. Compilation of valance and conduction bands for transition metal oxides(a);Organic semiconductor on an absolute scale (b) [37]

The table 1 shows metals and their work function.

Table 1: Metal and their work functions. [38]

Metal	Work Function (eV)
Aluminum	4.3
Gold	5.1
Chromium	4.5
Silver	4.64
Copper	5.0
In ₂ O ₃	4.3-5.0

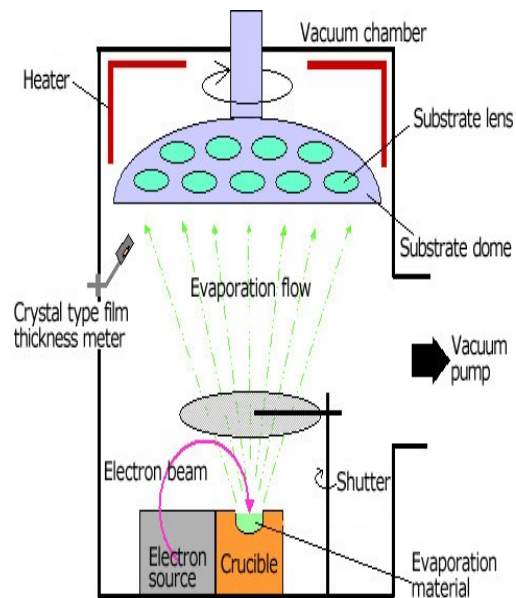


Figure 15. Vapor deposition chamber

Deposition of different materials using vapor deposition chamber for lateral and vertical structure diode. The substrate holder holds substrate and place in inverted position in the chamber. Electron gun heat the metal to its melting point and vaporize metal. In order to get desired pattern, shadow mask was used. The evaporation rate was 3As^{-1} and the vacuum level at 10^{-6} mbar. The metal vapor rises up and deposited. Different metals like gold, silver, aluminum, copper were deposited and measured. The thickness of deposited metal was 52nm and 100nm.

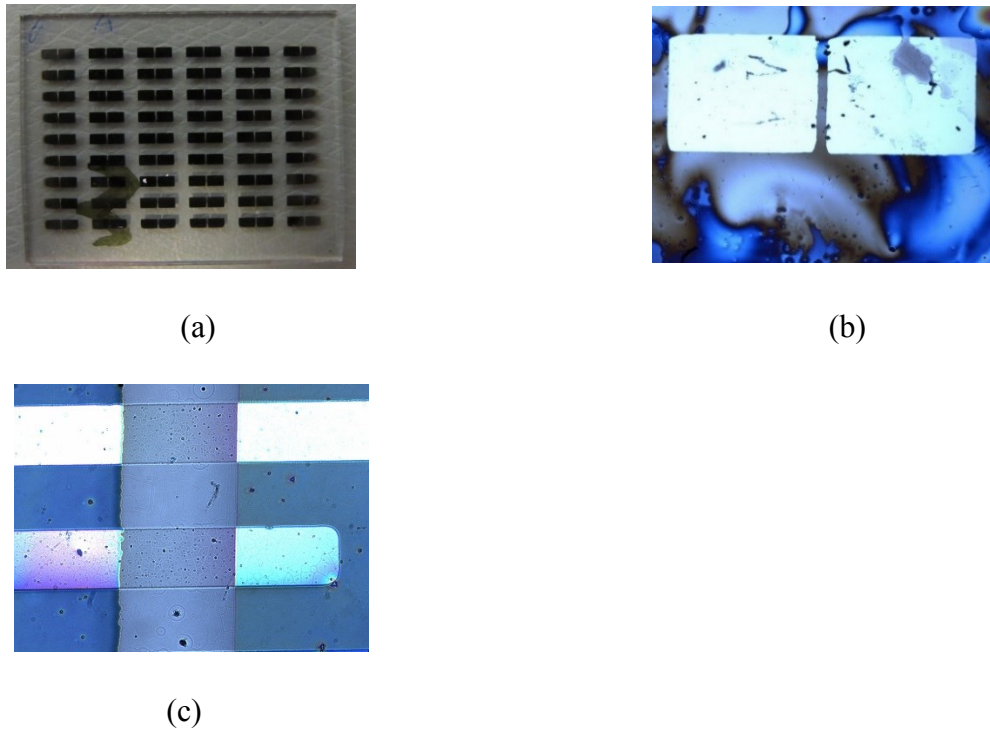


Figure 16. Vapor deposition; 0.4 M IO (a), 0.2M IO lateral (b), 0.2 M IO vertical (c)

The figure 16 (a) and (b) shows 0.4 molar solution of indium oxide spin coated on glass substrate and chromium deposited on it and p-type silicon wafer also used as substrate respectively. The deposition rate was 2 A s^{-1} . There was no proper deposition of chromium and observed peel off some layer on the glass. Similarly, there observed crater and island structure on silicon substrate and the deposition was not good,

In addition, for the vertical structure the aluminum as deposited as bottom electrode and chromium on the top. The deposition rate for aluminum and chromium varied because of their chemical properties. The deposition rate for aluminum was $3 \text{ A}^\circ\text{s}^{-1}$ and for chromium reduced to $0.5 \text{ A}^\circ\text{s}^{-1}$.

Table 2: Diode structure and deposition

Sample	Structure	Concentration	Metal	Thickness
1	Lateral	0.2M, 0.4M	Al	52nm
2	Lateral	0.2M	Cu	52 nm
3	Lateral	0.2M	Au	52 nm
4	Lateral	0.4M	Ag	100 nm
5	Lateral	0.2M, 0.4M	Cr	52 nm
6	Vertical	0.2M, 0.4M	AL	52 nm
7	Vertical	0.2M, 0.4M	Cr	100 nm
8	Vertical	0.2M, 0.4M	Pd	52 nm
9	Vertical	0.4M	Au	100nm
10	Vertical	0.4M	Al	100 nm
11	Lateral	0.4M	Ti	4 nm

The table 2 shows the vertical as well as lateral structure of the diode deposited using E-beam vapor deposition technique. The deposition were applied for 0.2 M and 0.4 M solution. Crucibles used were aluminum, copper, silver, gold, chromium, palladium etc. The different deposition rate used for different materials depending on the vaporization temperature. Some deposition rate that were done in the lab were 0.5 A°s-1, 2 A°s-1 and 3 A°s-1. High electron bombarding on the crucibles melts the material and deposited on the substrate.

3.4 Device structure

3.4.1 Lateral structure

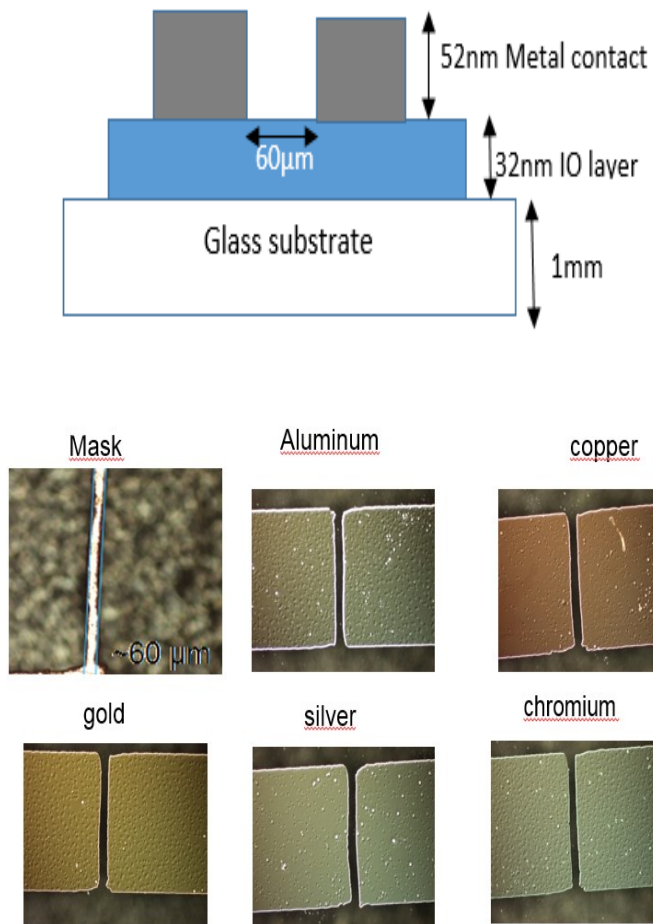
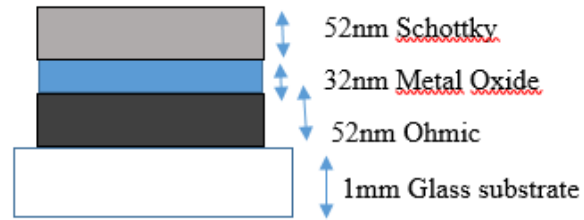


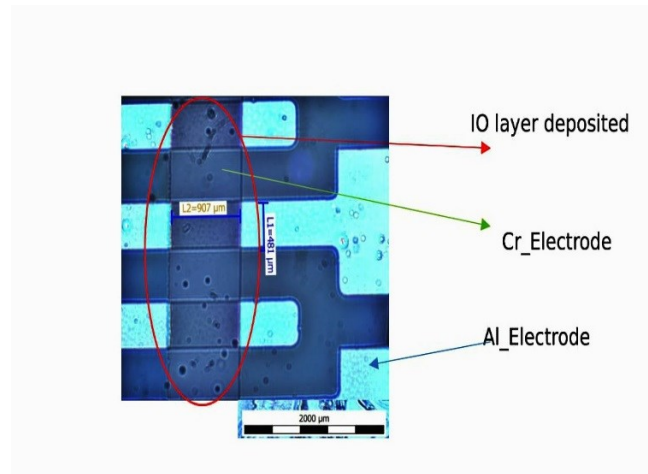
Figure 17. Lateral structure (a) ;Microscopic images of lateral contact(b)

Glass of 2cm * 2cm used as a substrate and indium oxide of thickness 32nm was deposited on it by spin coating. Then, vapor deposition method applied for the deposition of different metals on it. The thickness of metal contact deposited was 52nm. For the lateral structure, the distance between metal contacts is 60μm. Similar type metal contacts one acting as an anode and another as cathode. As different biasing voltage is applied flow of current from one electrode to another through the semiconducting layer. The area of lateral structure was vertical small to compare to vertical structure.

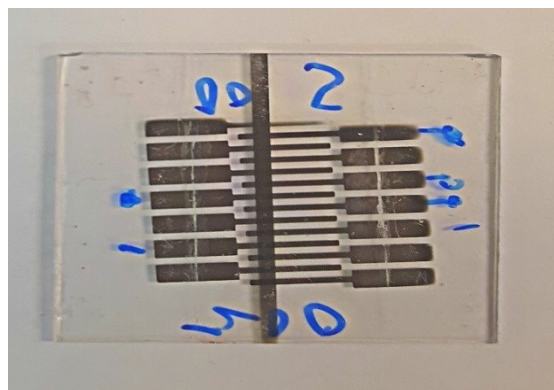
3.4.2 Vertical Structure



(a)



(b)



(c)

Figure 18. Vertical structure(a), microscopic view of diode(b), fabricated diode(c)

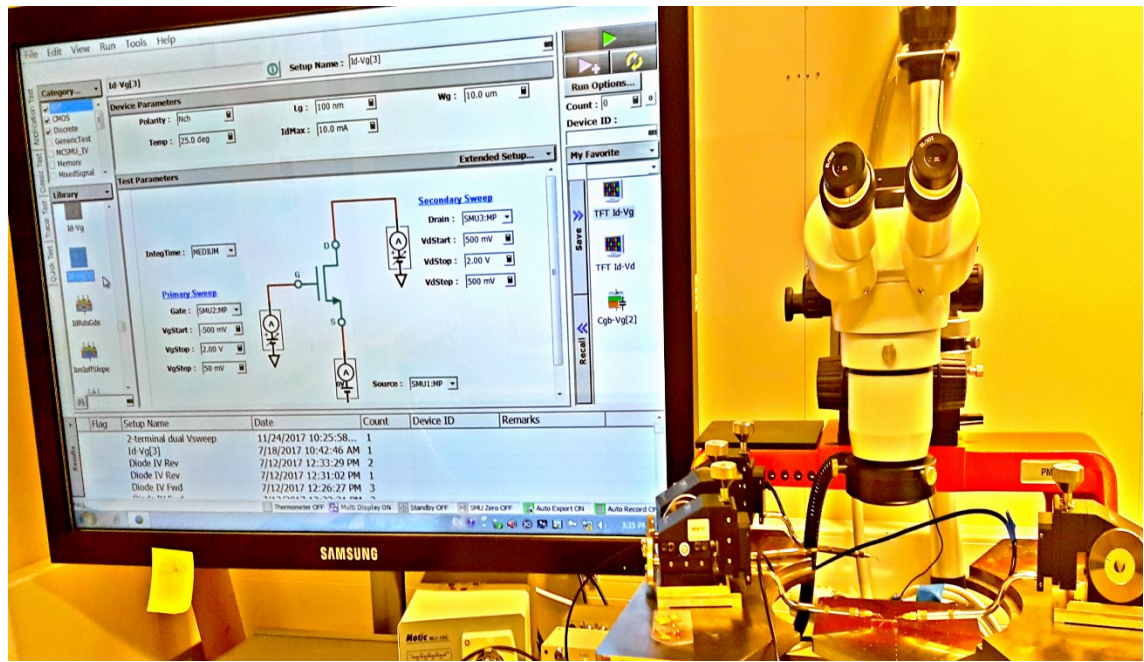
Similarly, for vertical structure two metal contacts were deposited one for Schottky and one for Ohmic in sandwiched slayer. E-beam deposition technique for the metal deposition. Aluminum, deposited on the top of substrate and metal oxide layer was spin coated on it. Then, chromium deposited on the top of semiconducting layer as shown in figure above. The thickness of aluminum and chromium layer was 52nm. Similarly, the thickness of metal oxide layer was 32nm. The mask was used area 1mm * 1mm. Initially, chromium expected as Schottky because of high work function and Aluminum as Ohmic contact. The device has an active area around 1mm². Optical Microscope, Olympus measure the dimension of diode.

3.4.3 Measurements

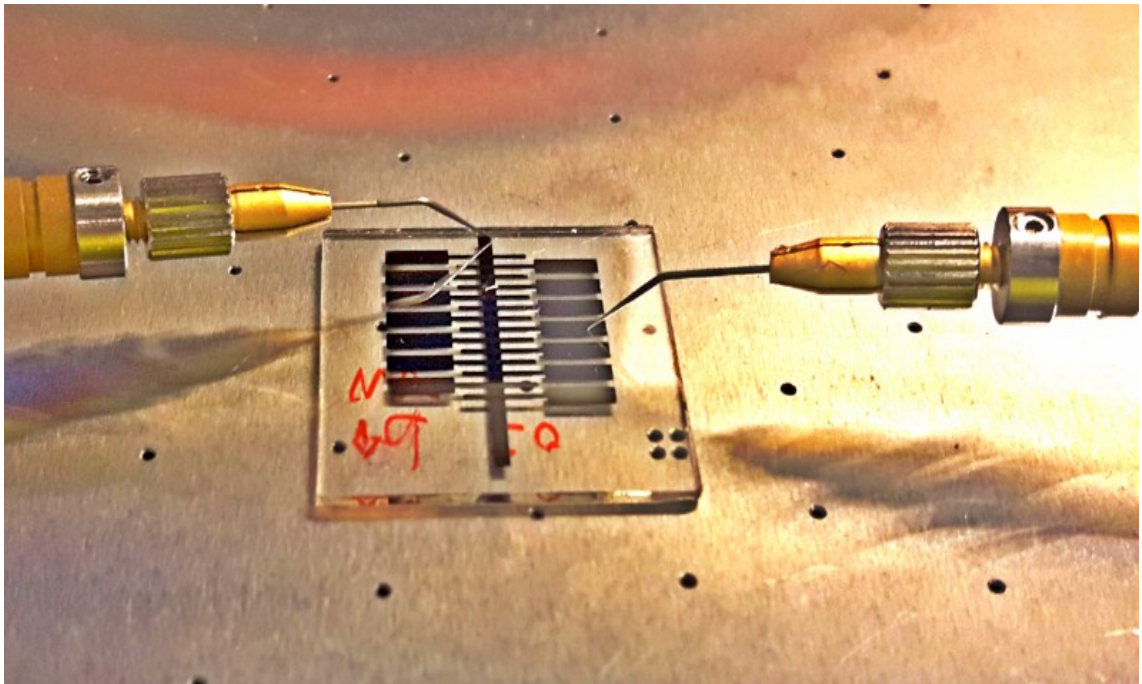
Different measurements set up were used to measure the device. Measurements are done at room temperature as well as inside glove box. They were Key sight semiconductor analyzer, Zahner potentiostatic measurements, temperature dependence measurement. The ORC temperature dependence consists of probe station attached with computer. The setup performed at different temperature range in probe station to see the temperature dependence of metal. That setup operates in two-electrode configuration. Coaxial cable and cantilever type mechanical probes for the electrical supply and connections. The use of analyzer and potentiostatic measurements helps to compare as well as analyze the results. It helps to determine whether the measurement process that is during the experiment is in right track or not. As, the samples are more sensitive to outside atmosphere, they were kept in glove box maintain oxygen, humidity and measured. There was a probe station inside the glove box and the cables connected to the potentiostatic setup unit. The measurements and different biasing voltages given below. The scan rate of voltage swept in both direction is 500 mVs⁻¹.

Table 3: Measurement unit at different biasing voltages

Measurement Unit	Biasing voltage
Potentiostatic	-20V to +20V
Key sight	-10V to +10V
ORC temperature dependence	-20V to +20V

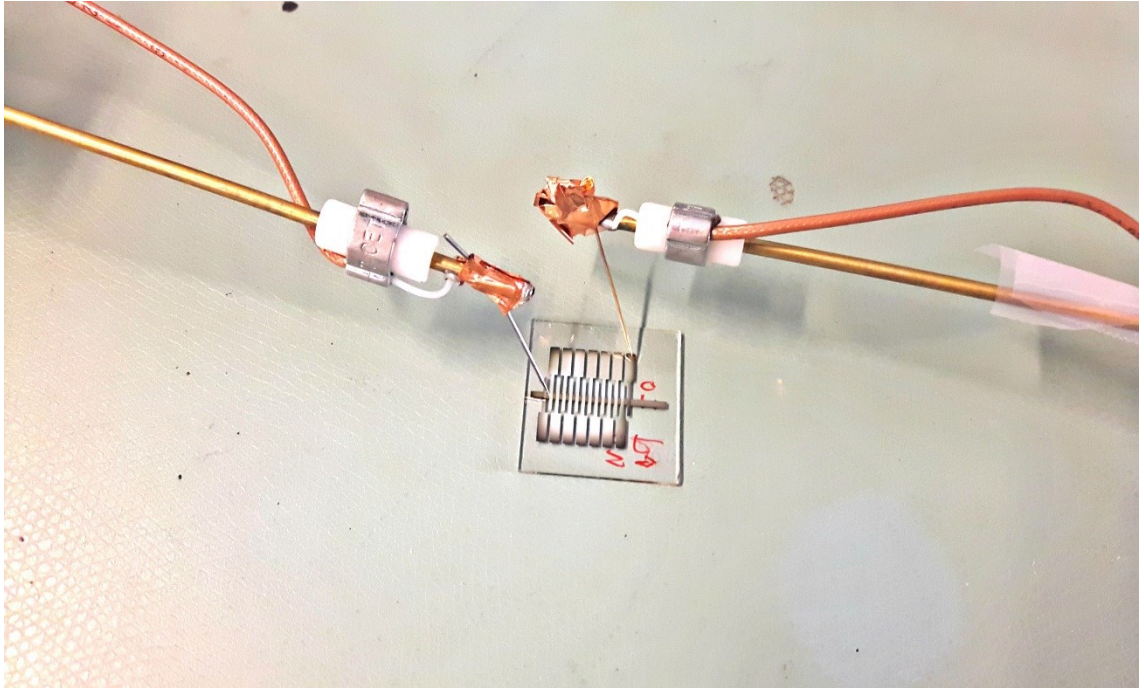


(a)

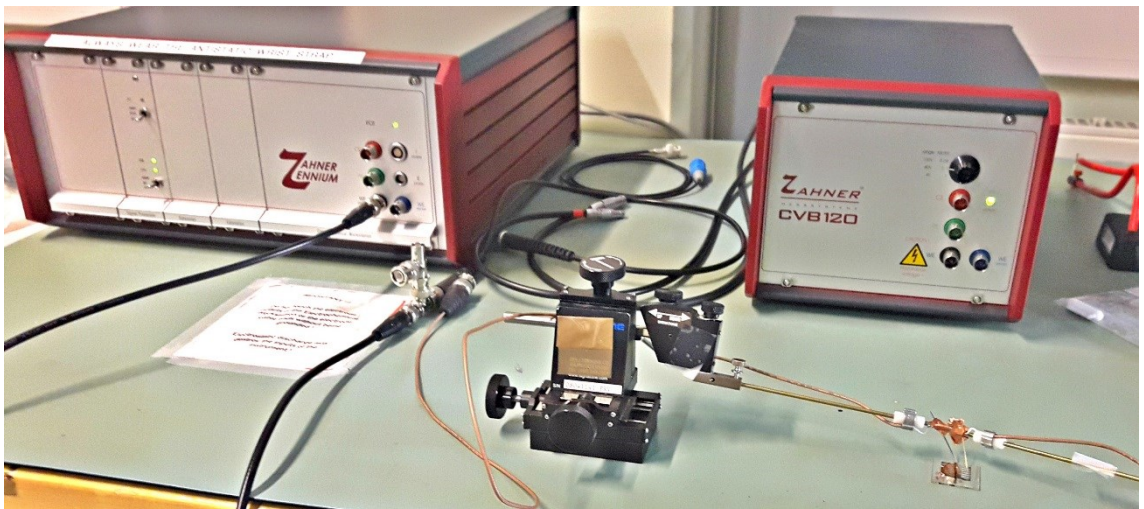


(b)

Initial measurements were to determine whether the metals are Ohmic or Schottky. The current–voltage characteristics were analyzed and measured for lateral and vertical structure diode at different biasing voltages. The comparison of results were done for the different measurement setup.



(c)



(d)

Figure 19. Measuring unit; Keysight(a,b) and Zahner(c,d)

4. RESULTS AND DISCUSSION

4.1 Semiconductor layer thickness

Optical profilometer was used for the measuring the thickness of the semiconducting layer on the glass substrate. The six number of samples fabricated at different RPM from 1000 to 6000 for 20 seconds. The thickness of samples those samples were measured many times with profilometer and average value for the thickness of the layer was obtained. The semiconducting layer was uneven and not uniform. Under the optical microscope, there can be observed craters structures, cracks, island etc. Table shows the values of the thickness at different RPM.

Table 3: Average thickness measurement results

Spin coating RPM at 20 seconds for samples	Semiconducting layer thickness (nm)
1000	45.466
1500	41.82
2000	32.18
3000	27.96
4500	22.89
6000	20.82

Layer thickness decreases as the spin coating parameter increases. The highest thickness was 45.466 nm at 1000 rpm and lowest thickness was 20.82nm at 6000 rpm. The thickness vary from sample to sample by 3-5 nm values.

The semiconducting thickness plays important role in current-voltage analysis of Schottky diode. A device with lower thickness gives higher current density than the device with higher thickness. Based on research paper, the current density becomes small, if layer is thick. In addition, there has be observed that the semiconducting thickness and series resistance are dependence on each other. [39] Magneto sputtering is also one of the technique for layer deposition on glass substrate. The desired layer can be deposited normally 25nm to 200nm. The break down voltage becomes higher if there is increasing thickness from 25nm to 200nm. [1]

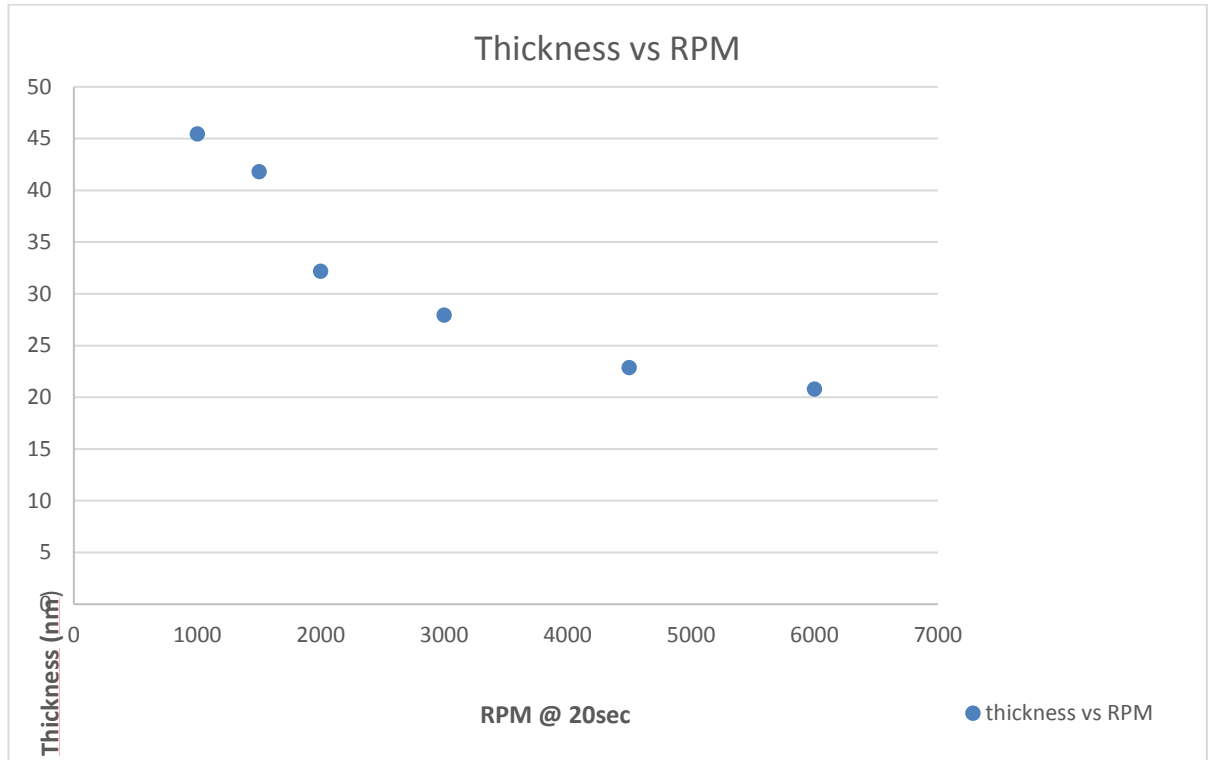


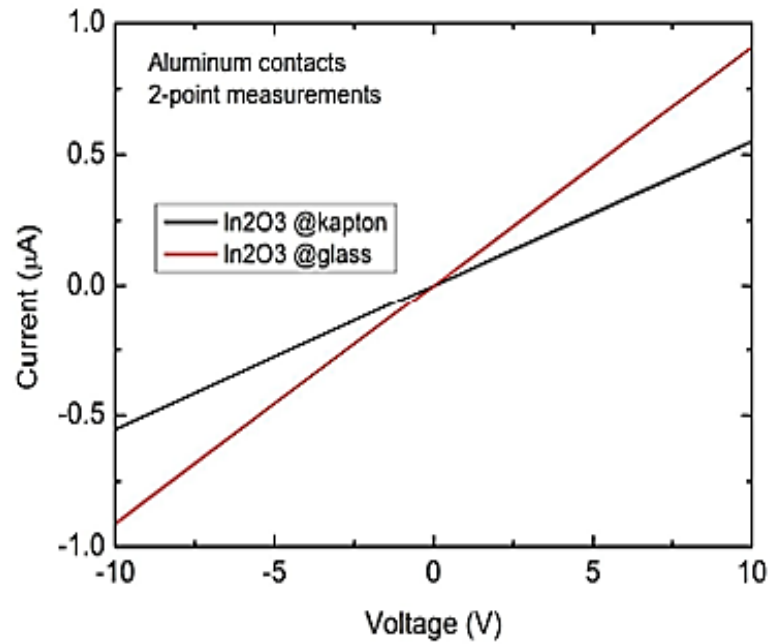
Figure 20. Thickness vs RPM

4.2 Metal contacts Current-Voltage analysis for Lateral structure

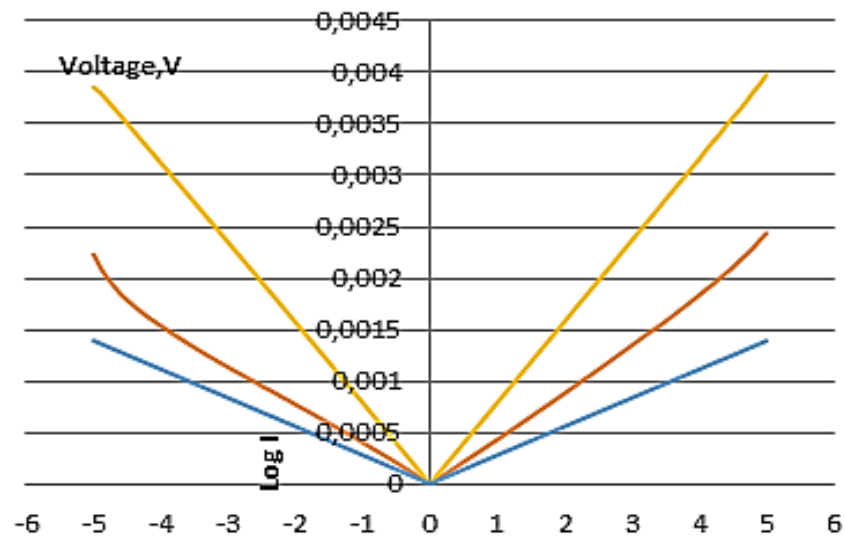
4.2.1 Aluminum

Aluminum metals were deposited on glass substrate and Kapton tape by vapor deposition. The metal contacts thickness was 50nm. And, semiconducting layer was around 32nm. The temperature dependence measurement was done in ORC and potentiostatic measurement was done at room temperature.

The current voltage characteristics for Aluminum shows Ohmic results as shown in figure 20 (a, b). Initially Aluminum was chosen for Ohmic with work function of 4.2 eV. The current-voltage curves have similar symmetry and are linear. [33]



(a)



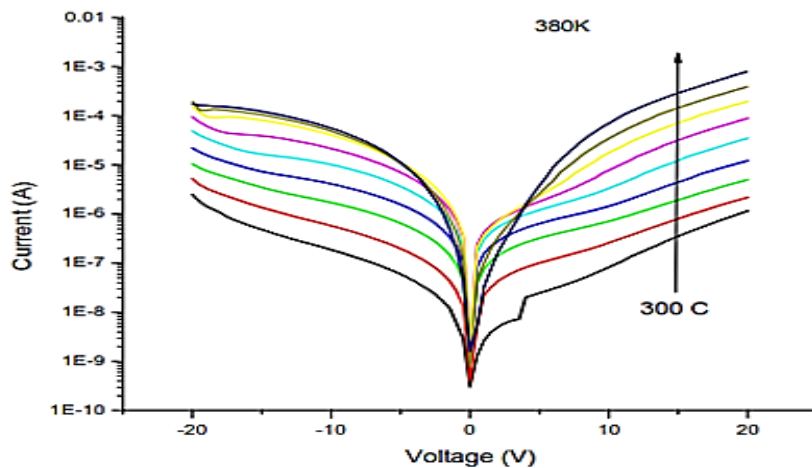
(b)

Figure 21. *I-V Characteristics of Aluminum(a, b)*

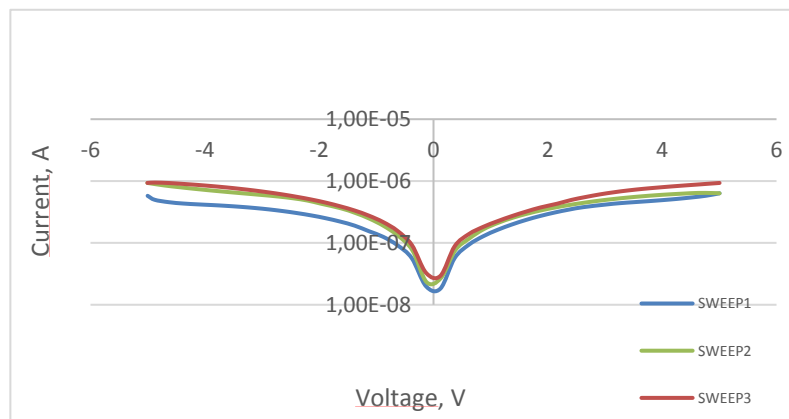
4.2.2 Chromium

Chromium was chosen for Schottky contact and having high work function 4.5 eV. It will form a large barrier on the semiconductor because of its high work function. Chromium is less refractory compared to nickel, gold, silver, copper, etc. as a result, electron beam deposition is easy for depositing chromium. [40] Temperature dependence measurements show that chromium is dependent on temperature. The temperature range was from 300 K

to 380 K by the steps increment of 20 K. Initially, the conduction was very low as the temperature increased from 300K to 380 K, it starts conducting. Symmetrical results on both sides in figure shows chromium does not work as Schottky as based on some experiments. The current value obtain in graph is also high, this might be because of pinholes, short circuits etc. Study of the temperature dependence regarding to current-voltage characteristics, related to Schottky diode will gives necessary ideas and results on barrier height, metal-semiconductor junction etc. [40]



(a)

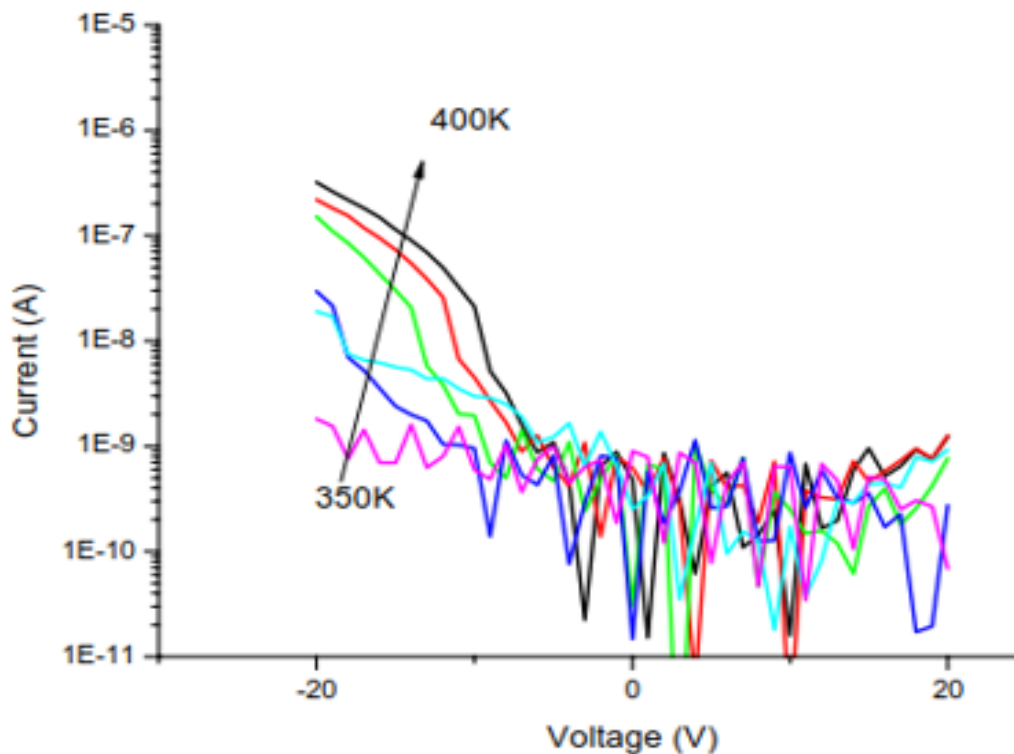


(b)

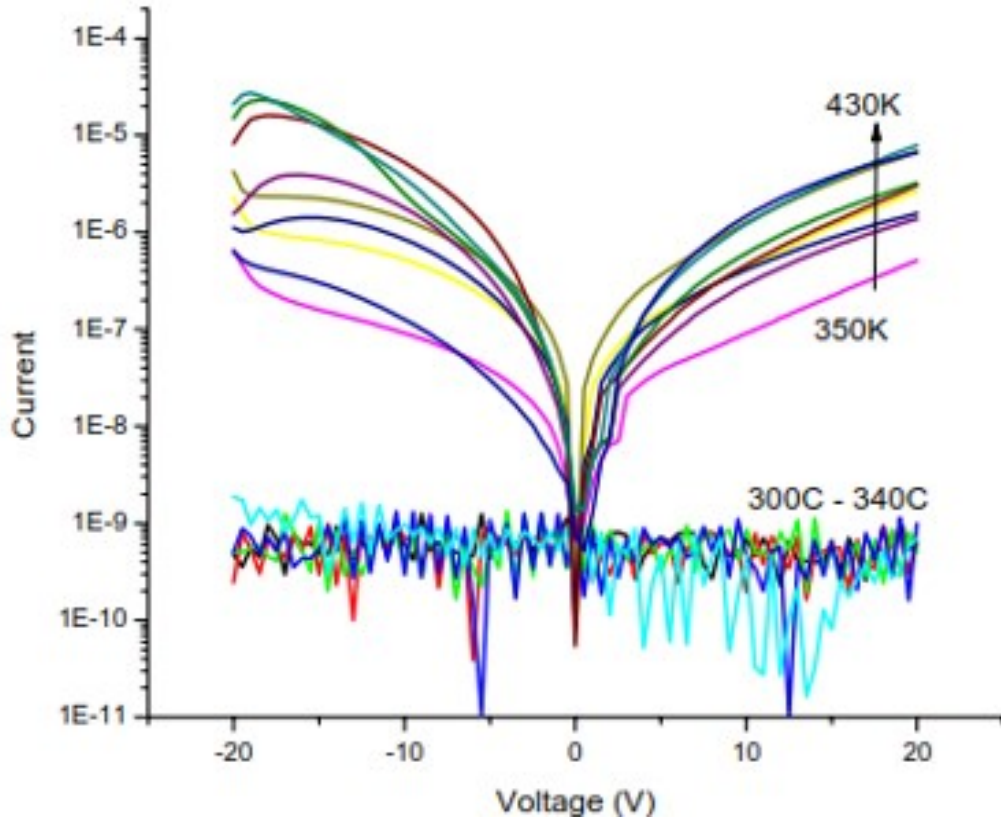
Figure 22. Temperature dependence and I-V characteristics of Chromium (a, b)

4.2.3 Silver and Gold

The work function of silver and gold are 4.64 eV and 5.1 eV respectively. Normally, silver and gold are used for Schottky contacts. So, in this experiment fabrication of lateral structure of gold and silver. Then, measured at biasing voltages from -20 V to +20 V. Silver with indium oxide starts conducting at higher temperature, which was 350 K to 400 K. Similarly, gold also start conducting at similar temperature as silver. The current voltage curves are not symmetrical and linear for silver. Values are random and fluctuating during biasing. Reverse current is much higher than the forward current in silver at 20 V. For gold after 350 K, current voltage are symmetrical. On the other hand, some research article shows, [H. Sheng, Applied physics letter, 2002] silver with zinc oxide film gives good Schottky contact at temperature of 295 K. The Schottky barrier heights was around 0.92 eV [41]. Similarly, gold formed good Schottky barrier with other semiconducting like n-InP, GaN. In the first case, the conducting temperature was in range of 60-300K, Mo used as Ohmic. [42] This temperature is lower comparison to temperature dependence test done shown in figure (b). In addition, in second case, Aluminum was used as Ohmic contact. [43]



(a)



(b)

Figure 23. Temperature dependence and I-V characteristics : Silver(a), Gold(b)

4.2.4 Copper

The work function of copper is 5.0 eV. Copper starts conducting, as temperature is greater than 320 K. Copper shows non-zero current behavior at when voltage is zero. Current - voltage curves are asymmetrical.

In this experiment, copper shows the temperature dependence at 310 K. However, based on article, [Ö. Demirciouglu,2011], when the Schottky barrier was formed between n-type silicon and copper germanide, current-voltage measurements was achieved at temperature range between 80 K to 300 K. [44] Similarly, for copper and silicide temperature was 95 K to 295 K. Comparing this case, the temperature range was high as shown in figure 24. This show copper with indium oxide has high temperature dependence comparing copper with germanide or silicide. [45]

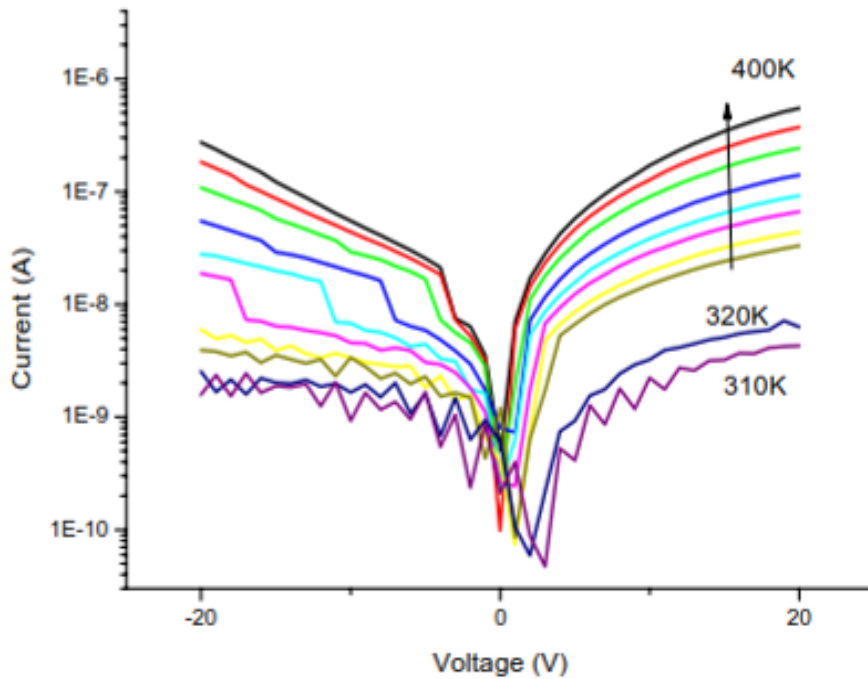
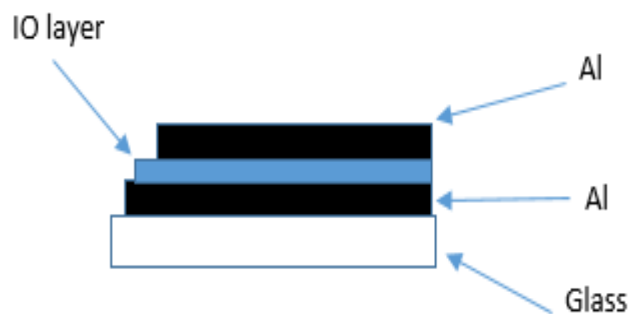


Figure 24. Temperature dependence and I-V characteristics of copper

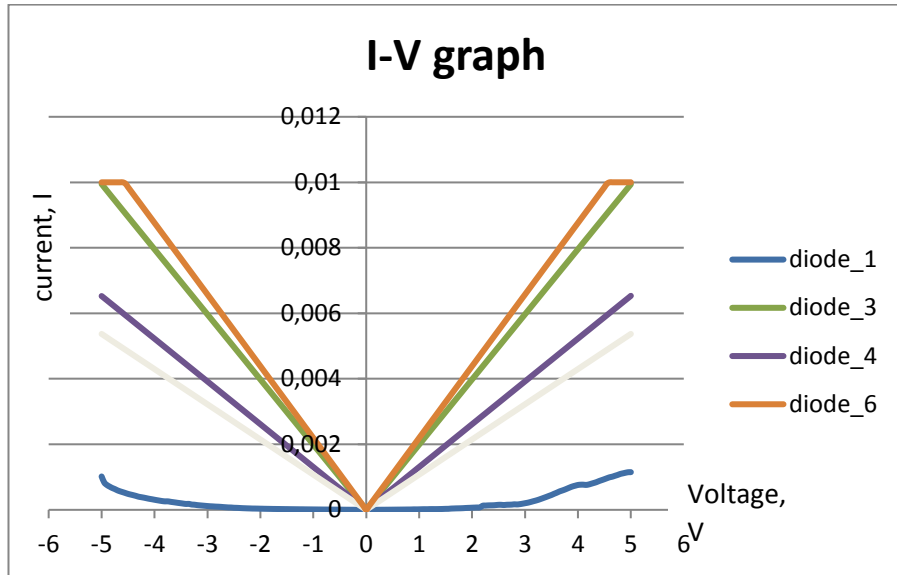
4.3 Metal contacts Current-Voltage analysis for Vertical Structure

4.3.1 Aluminum (Al/IO/AL)

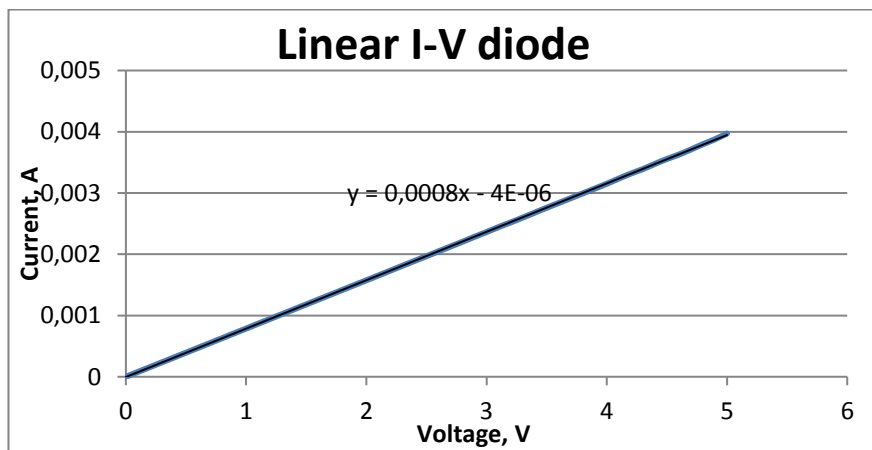
Vertical structure diode was fabricated using vapor deposition technique. Aluminum as both top and bottom electrode about the thickness of 50nm, Al/IO/Al. Thin layer of indium oxide was deposited between them about 32 nm.



(a)



(b)



(c)

Figure 25. Vertical structure and I-V characteristics of Al/IO/Al

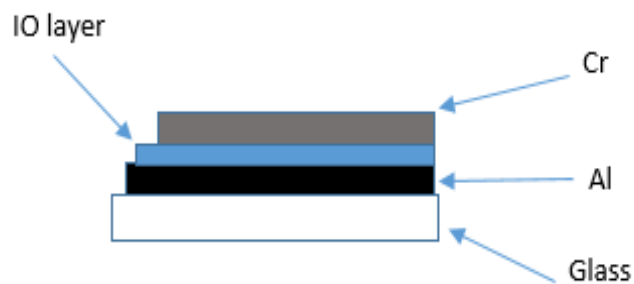
From the figure 25(a, b) it can be observed that the current-voltage characteristics are symmetrical and linear. The vertical diode structure of aluminum shows good Ohmic contact. [33] The current in forward bias and reverse bias are equal at biasing voltages from -5 to +5. In addition, the figure 25 b shows it follows equation of straight line $y = mx + c$ where, m is slope gradient and c is constant. [46]

The current obtain at 5V around 10 mA, which is very high. The turn on resistance is also low about 1250 ohm. This might be because of short circuit between electrodes. In addition, the spin coating was not uniform and smooth. Microscopic view shows cracks and defects. Based on research paper [47] for Al, N type regions must heavily doped to be

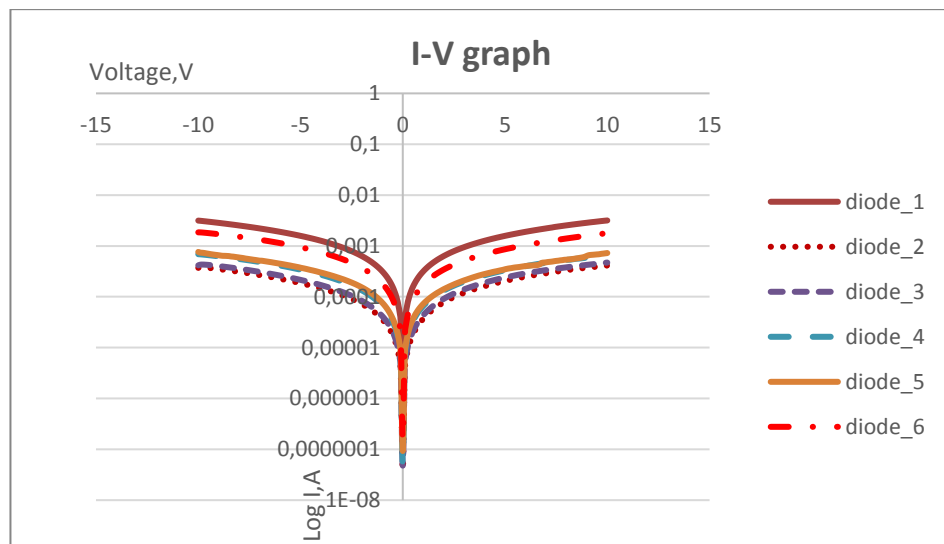
Ohmic. Similarly, Al need lightly doped to get Schottky contact. Al/IO/Al n-type gives Ohmic results in the experiment performed in this thesis.

4.3.2 Chromium (AL/IO/Cr)

The diode fabricated with vertical structure, Al as bottom electrode and Cr as top electrode. The thin interface layer of IO was between those metal. The thickness of metal contact and IO layer was 50 nm and 32 nm respectively. Figure 26 (a, b) shows vertical structure and current voltages characteristics. Key sight measuring unit at different biasing voltages is for electrical characterization..



(a)



(b)

Figure 26. Vertical structure (a), I-V characteristics (b)

The current density of fabricated diodes measured. Diode 1 gives maximum current of 3.16 mA. The current-voltage curves are symmetrical at different biasing voltages. Contacts shows Ohmic behavior. There are some defects on the semiconducting layer during preparation process. Cracks, pinholes, crater structure that might results short circuit of the contacts. In addition, some defects should be crystalized semiconducting layer or residual organic matter on the glass.

4.4 Current-voltage characteristics comparison of indium oxide and indium gallium oxide

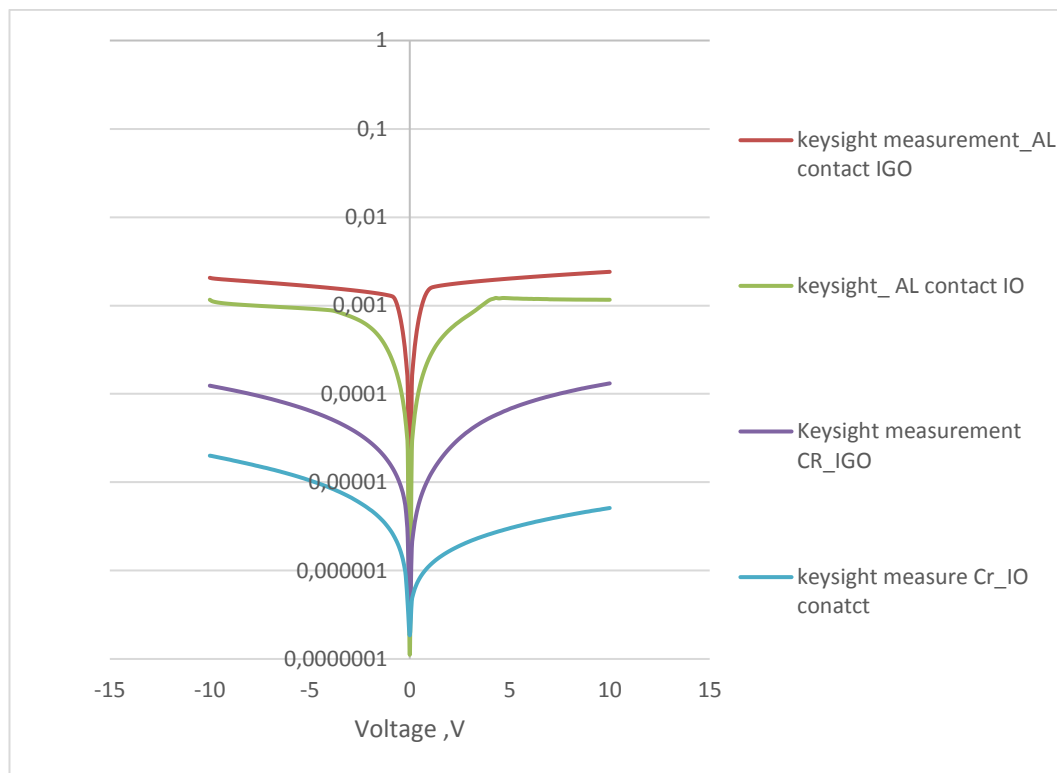


Figure 27. *I-V characteristics IO and IGO*

The lateral structure samples were fabricated using 0.2 M IO and 5%, 0.2M IGO. IO and IGO act as transparent semiconducting layer between the metal contacts. Two metal contacts fabricated were aluminum and chromium. Key sight measuring unit at different biasing voltages -10 V to +10 used to measure current-voltage characteristics. As shown in figure 27, the indium gallium oxide gives higher current mobility than the indium oxide.

Aluminum with indium gallium oxide have greater current than aluminum with indium oxide. The first one have 2.4 mA and second one have 1.1 mA. That is first one is two times higher second one. Similarly, chromium with indium gallium oxide also gives

higher current than chromium with indium oxide. The first one have 131 μA and second on have 5 μA . The current density difference is very high between them.

4.5 Current-voltage characteristics comparison glove box vs room temperature

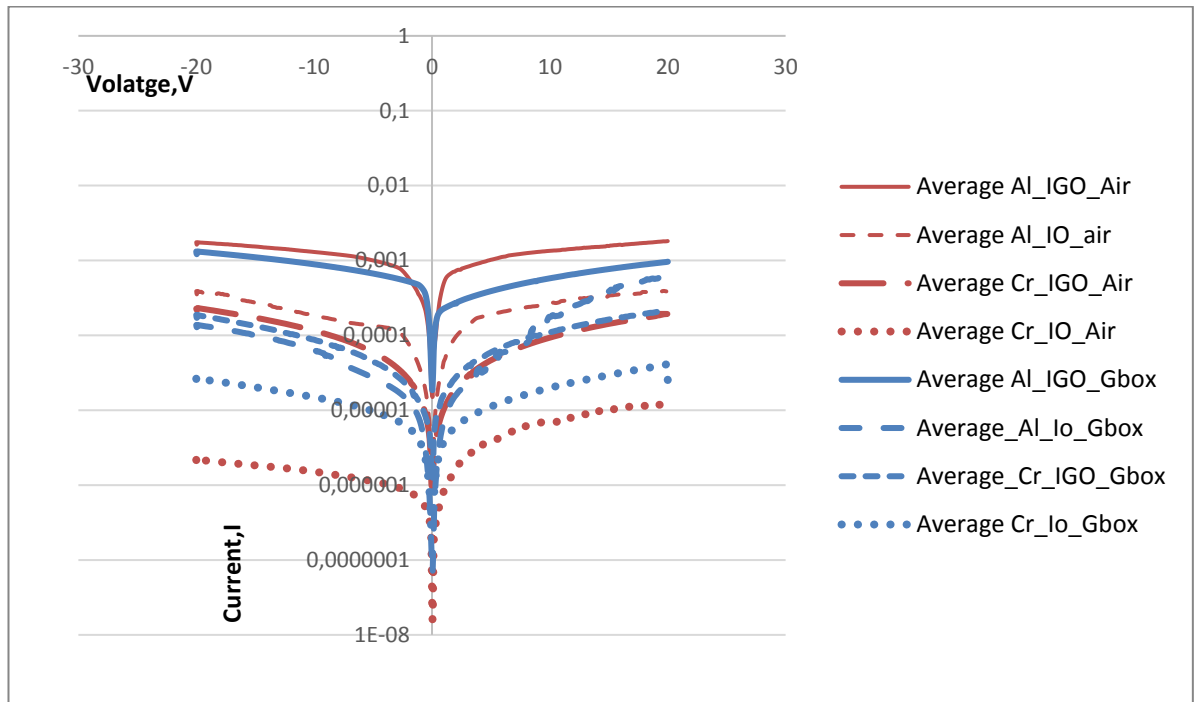


Figure 28. *I-V characteristics inside glovebox and outside area*

Measurements were done inside glove box as well as outside at room temperature. Aluminum and chromium were the metal contacts used for the semiconducting layer indium oxide and indium gallium oxide. Inside the glove box, the measuring probe station installed. The probe station that connected to Zanker measurement unit for electrical characterization. The average measurements vary and depends on areas, condition during measurement. The average current density was high inside glove box than outside area. Environmental conditions like temperature, pressure, vacuum etc. affect the measurement process. In addition, indium oxide and indium gallium oxide are very sensitive to external environment, which degrades the performance of the material. The average current obtained at maximum 20V shown in table below.

Table 4: Average current measured glove box vs room temperature measured +20V

Contacts_ measured	Average current measured at 20 V air, outside area	Average current measured at 20 V inside glove box
Al-IGO	1.8mA	963 μ A
Al-IO	396 μ A	639 μ A
Cr-IGO	193 μ A	218 μ A
Cr-IO	13 μ A	24,00 μ A

5. CONCLUSIONS

In this thesis, metal oxides used as a semiconducting layer between the metal contacts. The different metal contacts selected depending on their work functions. For examples metals like chromium, aluminum, copper, silver etc. studied and fabricated for both lateral as well as vertical structure. Different measurement unit used for the study of Schottky and Ohmic contact. Also, increasing speed of spin coater decrease the thickness of the semiconductor layer in substrate.

The temperature dependence measurements shows that silver and gold are temperature dependent between 350 K-450 K and starts conducting. Similarly, copper shows temperature dependence above 310 K. The measurement of lateral structure of diode shows that current-voltage characteristics are linear and symmetrical. The results are Ohmic. Even for vertical structure, the results are Ohmic instead of Schottky. This might be because indium oxide surface has electron accumulation layer that prevent the formation of rectifying Schottky contacts. UV ozone treatment done at different stages, which might help in good oxygen stoichiometry and metal-indium oxide layer have less defect density. This results in Schottky barrier. However, this technique also fails. The reason might be inhomogeneous spin coating, cracks, pinholes, crystalized indium oxide etc. The thickness of the semiconducting layer was also too small that might short circuit a diode.

Further experiments needs to be perform in future in order to achieve the Schottky contact. Increasing the thickness of semiconducting layer might help in pinholes free and prevent short circuit of diode. Schottky contact shadow mask for better understanding the contact resistance, indium oxide with different metal contacts. As indium oxide is very sensitive to external environment, the fabrication of diode should done inside the glove box and might give better results.

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